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**APPLICATION NUMBER: 60/546,680**

**FILING DATE: February 20, 2004**

**RELATED PCT APPLICATION NUMBER: PCT/US05/05574**

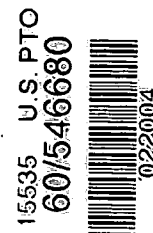


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PROVISIONAL APPLICATION FOR PATENT COVER SHEET  
Express Mail No. EV309119907US



|   |            |   |  |   |         |
|---|------------|---|--|---|---------|
|   |            | Docket Number   | 126457.1200  | Type a plus sign (+)<br>inside this box | +       |
| INVENTOR(s)/APPLICANT(s)  |            |   |  |   |         |
| LAST NAME   | FIRST NAME | MIDDLE INITIAL  | RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY) |   |         |
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|   |            |   |  |   |         |
|   |            |   |  |   |         |
| TITLE OF THE INVENTION (280 characters max)   |            |   |  |   |         |
| NON-POROUS ADHERENT INERT COATINGS AND METHODS OF MAKING  |            |   |  |   |         |
| CORRESPONDENCE ADDRESS  |            |   |  |   |         |
| PEPPER HAMILTON LLP<br>FIRM 21269<br>Attention: Raymond A. Miller<br>500 Grant Street, 50th Floor<br>Pittsburgh, PA 15219<br>(412) 454-5000 |            |   |  |   |         |
| STATE   | PA         | ZIP CODE  | 15219  | COUNTRY                                 | USA     |
| ENCLOSED APPLICATION PARTS (check all that apply)   |            |   |  |   |         |
| <input checked="" type="checkbox"/> Specification (Number of pages) [55]  |            | <input type="checkbox"/> CDs (number) [ ]   |  |   |         |
| <input checked="" type="checkbox"/> Drawings (Number of sheets) [6]   |            | <input checked="" type="checkbox"/> Other (specify): Postcard, Certificate of Mailing |  |   |         |
| METHOD OF PAYMENT (check one)   |            |   |  |   |         |
| <input checked="" type="checkbox"/> Applicant(s) claim(s) small entity status. See 37 C.F.R. § 1.27.  |            |   |  | PROVISIONAL FILING                      | \$80.00 |
| <input checked="" type="checkbox"/> A check or money order is enclosed to cover the Provisional filing fees                                 |            |   |  | FEE AMOUNT (\$)                         |         |
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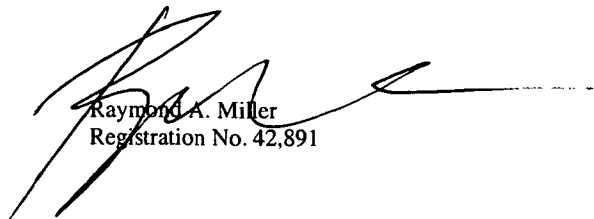
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.

☐ Yes, the name of the U.S. Government agency and the Government contract number are: \_\_\_\_\_

☐ Additional inventors are being named on separately numbered sheets attached hereto.

Respectfully submitted,  
PEPPER HAMILTON LLP

  
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# FEE TRANSMITTAL for FY 2004

Effective 10/01/2003. Patent fees are subject to annual revision.

☒ Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT (\$ ) 80.00

**Complete if Known**

|                      |                   |
|----------------------|-------------------|
| Application Number   | not yet assigned  |
| Filing Date          | February 20, 2004 |
| First Named Inventor | Niermeyer         |
| Examiner Name        | n/a               |
| Art Unit             | n/a               |
| Attorney Docket No.  | 126457.1200       |

**METHOD OF PAYMENT** (check all that apply)☒ Check ☐ Credit card ☐ Money Order ☐ Other ☐ None☒ Deposit Account:Deposit  
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50-0436

Pepper Hamilton LLP

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| Large Entity<br>Fee Code (\$) | Small Entity<br>Fee Code (\$) | Fee Description        | Fee Paid           |
|-------------------------------|-------------------------------|------------------------|--------------------|
| 1001 770                      | 2001 385                      | Utility filing fee     |                    |
| 1002 340                      | 2002 170                      | Design filing fee      |                    |
| 1003 530                      | 2003 265                      | Plant filing fee       |                    |
| 1004 770                      | 2004 385                      | Reissue filing fee     |                    |
| 1005 160                      | 2005 80                       | Provisional filing fee | 80.00              |
| <b>SUBTOTAL (1)</b>           |                               |                        | <b>(\$ ) 80.00</b> |

**2. EXTRA CLAIM FEES FOR UTILITY AND REISSUE**

| Total Claims       | Extra Claims | Fee from below | Fee Paid |
|--------------------|--------------|----------------|----------|
| Independent Claims | -20** =      | X              |          |
| Multiple Dependent | -3** =       | X              |          |

| Large Entity<br>Fee Code (\$) | Small Entity<br>Fee Code (\$) | Fee Description  |
|-------------------------------|-------------------------------|--|
| 1202 18                       | 2202 9                        | Claims in excess of 20                                     |
| 1201 86                       | 2201 43                       | Independent claims in excess of 3                          |
| 1203 290                      | 2203 145                      | Multiple dependent claim, if not paid                      |
| 1204 86                       | 2204 43                       | ** Reissue independent claims over original patent         |
| 1205 18                       | 2205 9                        | ** Reissue claims in excess of 20 and over original patent |

**SUBTOTAL (2)** (\$ ) 0

\*\*or number previously paid, if greater; For Reissues, see above

**FEE CALCULATION** (continued)**3. ADDITIONAL FEES**

Large Entity Small Entity

| Fee Code (\$) | Fee Code (\$) | Fee Description  | Fee Paid |
|---------------|---------------|--|----------|
| 1051 130      | 2051 65       | Surcharge - late filing fee or oath  |          |
| 1052 50       | 2052 25       | Surcharge - late provisional filing fee or cover sheet                     |          |
| 1053 130      | 1053 130      | Non-English specification  |          |
| 1812 2,520    | 1812 2,520    | For filing a request for <i>ex parte</i> reexamination                     |          |
| 1804 920*     | 1804 920*     | Requesting publication of SIR prior to Examiner action                     |          |
| 1805 1,840*   | 1805 1,840*   | Requesting publication of SIR after Examiner action                        |          |
| 1251 110      | 2251 55       | Extension for reply within first month                                     |          |
| 1252 420      | 2252 210      | Extension for reply within second month                                    |          |
| 1253 950      | 2253 475      | Extension for reply within third month                                     |          |
| 1254 1,480    | 2254 740      | Extension for reply within fourth month                                    |          |
| 1255 2,010    | 2255 1,005    | Extension for reply within fifth month                                     |          |
| 1401 330      | 2401 165      | Notice of Appeal   |          |
| 1402 330      | 2402 165      | Filing a brief in support of an appeal                                     |          |
| 1403 290      | 2403 145      | Request for oral hearing   |          |
| 1451 1,510    | 1451 1,510    | Petition to institute a public use proceeding                              |          |
| 1452 110      | 2452 55       | Petition to revive - unavoidable   |          |
| 1453 1,330    | 2453 665      | Petition to revive - unintentional   |          |
| 1501 1,330    | 2501 665      | Utility issue fee (or reissue)   |          |
| 1502 480      | 2502 240      | Design issue fee   |          |
| 1503 640      | 2503 320      | Plant issue fee  |          |
| 1460 130      | 1460 130      | Petitions to the Commissioner  |          |
| 1807 50       | 1807 50       | Processing fee under 37 CFR 1.17(q)  |          |
| 1806 180      | 1806 180      | Submission of Information Disclosure Stmt                                  |          |
| 8021 40       | 8021 40       | Recording each patent assignment per property (times number of properties) |          |
| 1809 770      | 2809 385      | Filing a submission after final rejection (37 CFR 1.129(a))                |          |
| 1810 770      | 2810 385      | For each additional invention to be examined (37 CFR 1.129(b))             |          |
| 1801 770      | 2801 385      | Request for Continued Examination (RCE)                                    |          |
| 1802 900      | 1802 900      | Request for expedited examination of a design application                  |          |

Other fee (specify)

\*Reduced by Basic Filing Fee Paid

**SUBTOTAL (3)** (\$ ) 0**SUBMITTED BY**

(Complete if applicable)

|                   |                   |                                   |                   |           |              |
|-------------------|-------------------|-----------------------------------|-------------------|-----------|--------------|
| Name (Print/Type) | Raymond A. Miller | Registration No. (Attorney/Agent) | 42,891            | Telephone | 412.454.5000 |
| Signature         |                   | Date                              | February 20, 2004 |           |              |

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APPLICANT: NIERMEYER  
TITLE: NON-POROUS ADHERENT INERT COATINGS AND  
METHODS OF MAKING  
SERIAL NO.: NOT YET ASSIGNED  
ATTORNEY REF: 126457.1200  
DATE OF DEPOSIT: FEBRUARY 20, 2004  
EXPRESS MAIL NO. EV309119907US

I HEREBY CERTIFY THAT THIS PROVISIONAL PATENT APPLICATION IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE VIA UNITED STATES POST OFFICE EXPRESS MAIL UNDER 37 C.F.R. § 1.10 ON THE DATE INDICATED ABOVE AND IS ADDRESSED TO THE MAIL STOP PROVISIONAL APPLICATION, COMMISSIONER FOR PATENTS, P.O. BOX 1450, ALEXANDRIA, VA 22313-1450.

KATHLEEN PUJOL

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**DOCUMENTS ENCLOSED:**

1. New Provisional Application Cover Sheet (1 sheet);
2. Provisional Application (55 pages);
3. Formal Drawings (6 sheets)
4. Fee Transmittal and check in the amount of \$80.00; and
5. Certificate of Mailing;
6. Postcard.

# **NON-POROUS ADHERENT INERT COATINGS AND METHODS OF MAKING**

## **BACKGROUND AND SUMMARY**

[0001] There is a continuous effort to develop pressure sensors, chemical sensors, and housing materials that are lower in cost and smaller in size, yet are characterized by high reliability, sensitivity, and linearity. For example, multiple pressure sensors, temperature, and optical sensors having sensing diaphragms, cavities, and resistive elements can be made on a single silicon wafer using semiconductor fabrication processes. In the processing of such cells, sensor elements such as the thin diaphragm of a pressure sensor are formed in a silicon wafer through preferential chemical etching. Ion implantation and diffusion techniques are then used to drive doping elements into the diaphragm, forming resistive bridge circuit elements whose electrical resistance changes with strain. As a result, deflection of the diaphragm causes a change in resistance value of the piezoresistive elements, which can then be correlated to the magnitude of the pressure applied to the diaphragm.

[0002] The use of standard single-crystal silicon wafers and standard semiconductor device fabrication processes allows many such cells to be fabricated from a single wafer, providing some economy of scale. However, silicon is susceptible to chemical attack, particle generation, and erosion by various media, particularly in applications where a high-pressure, temperature, and corrosive fluids are to be sensed, e.g., semiconductor manufacturing, long term medical implants, and automotive applications. One particularly difficult and sensitive application of integrated circuits, semiconductors and metal electrodes is in electrical or electronic device implantation in a human or animal body. Extra-cellular fluids within the body are saline, and often contain a number of other ions or other electrolytes. At body temperatures,

are saline, and often contain a number of other ions or other electrolytes. At body temperatures, severe and rapid corrosion may lead to rapid and untimely failure of the device. For such applications, a pressure, temperature, or chemical sensor must also be of high chemical purity, physically rugged and resistant to the hostile environment of the sensed medium, necessitating that a micromachined silicon sensor cell include some form of protection in order to realize its advantageous operational characteristics in the chemically hostile environment. Current methods for producing media-compatible, high-pressure sensors include enclosing a silicon sensing chip in an inert fluid, such as a silicone oil or gel, and then further separating the sensing chip from the medium to be sensed with a metal diaphragm, such that pressure must be transmitted through the metal diaphragm and fluid to the sensing chip. While achieving some of the operational advantages of silicon pressure transducer cells, the manufacturing processes for these sensors are relatively expensive and complicated.

[0003] Piezoelectric and capacitive pressure sensors are typically thin ceramic plates or diaphragms that may be coated with thick-film electrodes or bridge elements to form capacitors or strain sensing elements. However, each of these also have certain disadvantages, such as complex circuitry to detect capacitance changes, the requirement for ceramic-to-ceramic bonds, and a maximum pressure capability typically not exceeding about 1000 psi (about 7 MPa). For higher pressures, metal diaphragms have found use as the sensing element, however these diaphragms are not generally useful in corrosive aqueous solutions. Metal diaphragms generally deflect more for a given thickness and pressure than ceramic diaphragms. With metal diaphragms bridge elements or electrodes may be deposited on to a dielectric insulating layer on the metal followed by thin-film polysilicon or metal deposited on the metal diaphragm to form the bridge or electrode structures. For example, a thin-film polysilicon layer is deposited on the

dielectric to form the piezoresistors of the bridge, followed by thin-film metallization to provide electrical interconnects. As is conventional, the thin-film layers are typically deposited by such processes as chemical or physical vapor deposition. The equipment necessary for these processes is expensive, and deposition rates are extremely slow. Deposition of the thin-film layers requires multiple patterning, exposure, developing and stripping steps for the required thin-film photoresists and metallization, and must be carried out in a controlled environment to assure that no air borne particles are present on the surface to be coated. In addition, because such processes deposit thin-films usually no thicker than 10,000 angstroms, the surface of the metal diaphragm must be extremely smooth to avoid rough surface features penetrating through or producing discontinuities in the deposited thin films. Finally, the resistance of the resulting polysilicon thin-film piezoresistors can vary dramatically with temperature.

**[0004]** Typically, a sensor is contained within a chemically and mechanically protective housing. The housing essentially surrounds the sensor and any associated electronics for sensor excitation and signal processing. While this provides mechanical protection for the sensor, protection from hazardous chemicals and contaminants in the medium must also be provided. In one type of pressure sensor assembly, a silicone gel, fluorosilicone gel, or silicone oil is applied over the external surface of a pressure sensor and essentially partially fills the housing in which the pressure sensor is mounted. The gel or oil is covered with a membrane. The manufacture of these cells can be cumbersome and expensive.

**[0005]** Various materials have been developed to provide an electrically insulative moisture barrier over a substrate. Among the more prominent of these are aromatic polyimides such as those sold under the trade designation "Kevlar" by E.I. DuPont de Nemours, & Co. However, polyimides are highly viscous, difficult to deposit, and can easily entrain gas bubbles



leading to film defects. Parylene N coatings are produced by vaporizing a di(p-xylylene) dimer, pyrolyzing the vapor to produce p-xylylene free radicals, and condensing a poly-oligomer from the vapor onto a substrate that is maintained at a relatively low temperature, typically ambient or below ambient. Parylene N is derived from di(p-xylylene), while parylene C is derived from di(monochloro-p-xylylene), and parylene D is derived from di(dichloro-p-xylylene).

[0006] Although parylenes have generally advantageous electrical, chemical resistance and moisture barrier properties, it has been found that these poly-oligomers do not adhere well to many substrate surfaces, particularly under wet conditions. Although these poly-oligomers are quite resistant to liquid water under most conditions, they are subject to penetration by water vapor which may condense at the interface between the parylene film and the substrate, forming liquid water which tends to delaminate the film from the substrate. Vapor deposited parylene films are also generally quite crystalline and are subject to cracking which may also create paths for penetration of moisture to the substrate surface.

[0007] These organic coatings may be used either alone or together with fluorosilicone gels. Fluorosilicone gels are used to protect the sensor device, wirebonds, portions of the package, and leads. Fluorosilicone gels have several disadvantages including an incompatibility with fuels (e.g., swelling).

[0008] This invention relates to protective coatings on sensor and optical substrates, and more particularly to a novel process for providing a tough, adherent, insulating coating that constitutes a barrier against penetration of fluids and ions to the surface of the sensor or electrodes and other structure deposited on the sensor.

[0009] Embodiments of the present invention include a structures having an effective thickness of a protective non-porous coating that is adherent and chemically bonded to a surface

of the structure in contact with a fluid. Structures coated may be but not limited to a sensor, a conduit, a vessel such as a cuvette or gas sampling cell, bellows, handling equipment, or transparent windows that are to be contacted with a fluid during chemical processing and preferably a fluid whose properties are to be measured or characterized. The protective and adherent coating is chemically inert, has low permeability to fluids, ions and gases. The adherent non-porous coating on a surface of the sensor protect it and structures formed on such as electrodes and electrical structures from corrosion, particle generation, delamination, or changes in sensor response and sensitivity caused by the fluid. The coating is prepared from a coating material that includes a solvent and a poly-oligomer or poly-oligomer that contains fluorine and reactive groups for chemically bonding a portion of the poly-oligomer to the sensor surface. The adherent coating can be formed by evaporation of solvent from a bubble free volume of coating material deposited onto the sensor surface to form a non-porous film. The non-porous film is cured to form the protective non-porous coating; the coating adheres to the substrate surface and at least a portion is chemically bonded to the surface of the sensor. The curing preferably occurs at a temperature less than the boiling point of the coating material solvent(s) and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. The cured film having a density, thickness and mass which permits said sensor to produce a measurable physical response to energy input to said sensing device and protects the sensor from particle generation, degradation, and corrosion especially in high purity corrosive fluids.

[0010] One embodiment of the present invention is a sensor having a surface with an effective thickness of an adherent protective coating on the surface of the sensor for contacting a

fluid to be measured. The property of the fluid to be measured may include but is not limited to pressure, flow, temperature, chemical composition, chemical purity, or a combination of these. The coating preferably results in an adherent non-porous coating on a surface of the sensor which protect it and structures formed on it from corrosion, particle generation, delamination, or changes in sensor response and sensitivity caused by the fluid. The coating includes a soluble poly-oligomer containing fluorine, that is chemically bonded to the sensor surface to form an adherent protective coating. The adherent coating is formed by curing a non-porous evaporated film of the coating material formed or placed on the surface of the sensor. Preferably the curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. The device and sensor may include structures for probing by electrical energy, light energy, mechanical energy, or chemical interaction the state of the sensor and means or structures for measuring said physical response of said sensor to the excitation. Such structure on one or more of its surfaces may include but are not limited to resistive, capacitive, transistors, electrical contacts, optical contacts, or a combination of these. The sensor includes a ceramic material. Preferably the non-porous film of the coating material formed or placed on the surface of the sensor includes greater than 10% of the poly-oligomer in the coating material deposited on the substrate to form the non-porous film. The sensor may be used to measure one or more fluid properties such as but not limited to temperature, flow, chemical purity, pressure, or a combination of these. The poly-oligomer coating material deposited or applied to the sensor surface can include an organosilane adhesion promoter. The adherent protective coating can be

formed on one or more surfaces of the sensor. Preferably the adherent protective coating is at least 50 micron thick. The sensor surface can be treated to chemically bond the poly-oligomer to the sensor surface. The poly-oligomer may bond to surface groups that includes but are not limited to reactive amine, hydroxyl, carboxylic, ester, amide, or thiol groups for bonding the fluorine comprising poly-oligomer to the sensor surface.

[0011] Another embodiment of the present invention is a method for measuring a fluid property such as pressure, flow, temperature, chemical composition, chemical purity, or a combination of these in for example a fluid sample or fluid in a conduit. The method includes contacting fluid with a sensor having a surface with an effective thickness of an adherent protective coating on the surface of the sensor for contacting the fluid to be measured. The adherent coating including a soluble poly-oligomer containing fluorine where a portion of the poly-oligomer is chemically bonded to the sensor surface to form an adherent protective coating. The adherent coating is formed by curing a non-porous film of the coating material formed or placed on the surface of the sensor. Preferably curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. Applying energy to the adherent coated sensing device, measuring the physical response of the sensor, and comparing the physical response to a standard relationship allows correlation of the coated sensor's physical response to a property of the fluid such as but not limited to pressure, temperature, purity, flow or a combination of these. The method can be performed using coated a sensor that is a piezoresistive, piezoelectric, or thermoresistive material. The method may be performed using a coated sensor that includes one

or more plates in capacitive sensor, having two plates and the adherent non porous perfluorinated coating is positioned on one of said plates on a surface in contact the fluid.

[0012] Another embodiment of the present invention is a method of making a coated sensor that includes forming a non-porous film of a coating material deposited on a chemically bondable fluid contacting surface of a sensor. The non-porous film of coating material includes a soluble poly-oligomer containing fluorine and reactive groups on the poly-oligomer. At least a portion of the poly-oligomer reactive groups are chemically reacted with groups on the surface of the sensor for bonding at least a portion of the poly-oligomer to the chemically bondable fluid contacting surface of the sensor. The non-porous film on the chemically bondable fluid contacting surface of the sensor includes greater than 10% of the poly-oligomer deposited to form the non-porous film, and preferably little or no coating material is wasted. The non-porous film may be formed by evaporation of the solvent from the coating material deposited on the sensor, alternatively a film of non-porous material may be placed on the sensor surface. The non-porous film of the coating material is cured on the chemically bondable fluid contacting surface of a sensor with the curing chemically bonding the poly-oligomer reactive groups to the chemically bondable surface of the sensor surface to form an adherent protective coating on the surface of the sensor. The non-porous film of the coating material on the chemically bondable fluid contacting surface of the sensor is formed by removing solvent from a solution of the poly-oligomer contacting surface of the sensor. The curing step preferably occurs at a temperature below which damage to resistive structures, capacitors, transistors, electrical contacts, optical contacts, or a combination of these results. Preferably curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater

than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. Even more preferably the curing is at a temperature of less than about 120°C. The method may be performed where the fluorine containing poly-oligomer includes an aliphatic ether ring in the chain of the poly-oligomer. In the method the fluid contacting surface can be cleaned and made chemically bondable by a treatment chosen such as but not limited to plasma etching, chemical modification with an adhesion promoter, oxidation, or hydroxylation of a surface. The sensor used in the method may include on one or more of its surfaces structures such as but not limited to resistor bridges, resistors, capacitors, electrodes, transistors, electrical contacts, optical contacts, bonding pads or a combination of these. The coating material may include an organosilane adhesion promoter to react with the sensor surface and also the reactive groups in the poly-oligomer to bond at least a portion of the poly-oligomers to the sensor surface to form an adherent protective film on the sensor surface. The method may be used to form an adherent protective coating on the surface of the sensor has a thickness of greater than 50 microns, greater than 100 microns, greater than 2,500 microns and multiple coatings may be deposited and cured. The method of coating may be used to coat a sensor diaphragm. The method may have the sensing means or structures such as but not limited to electrodes, resistive bridge structures, thermistors, optical and electrical input and output connection formed on a surface of the sensor prior to coating and prior to curing with the coating material. The method may involve solvent removal from coating material applied to the sensor surface to form the non-porous film where the evaporation is performed in an antistatic environment. The method may use curing that can be but is not limited to chemical curing, thermal curing, photochemical curing or a combination of these.

[0013] Another embodiment of the present invention is a sensing device for measuring the property of a fluid such as but not limited to pressure, flow, chemical purity, chemical composition, or a combination of these. The device includes a sensor having a surface with an effective thickness of an adherent protective coating on a surface of the sensor that is used for contacting a fluid to be measured. The coating includes a soluble poly-oligomer containing fluorine, where at least a portion of the poly-oligomer is chemically bonded to the sensor surface to form an adherent protective coating. The adherent coating can be formed by curing a non-porous evaporated film of the coating material formed or applied on the fluid contacting surface of the sensor. The device includes structures or means for applying energy to sensing device for excitation and means or structures for measuring the physical response of the sensor to the excitation and the fluid. The device includes a housing which isolates the fluid contacting and coated side of the sensor from the interior of the housing. The housing prevents fluid communication between the non-porous adherent film of the coating material formed on the fluid contacting surface of the sensor from a second fluid isolated surface of the sensor which may have circuit elements, bonding pads, and other structures deposited onto it. The device can have a sensor with a formed adherent protective coating that has a surface energy of less than 30 dynes/cm. The housing used to mount one or more sensors may include fluid inlet and fluid outlet connections. The device may include sensor materials which include but are not limited to piezoresistive, piezoelectric, thermoresistive or a combination of these properties. Preferably the device has a sensor where the non-porous (evaporated) film of the coating material formed on the surface of the sensor includes greater than 10% of the poly-oligomer in the coating material deposited on the sensors to form the film; little or no coating material is lost during the deposition and non-porous film forming process. Even more preferably, nearly all or, 100% of

the poly-oligomer in the coating material that is deposited on the sensor to form the coating film remains on the sensor surface; no reclamation or recycling of valuable perfluorinated coating materials or coupling agents etc. is needed, nor are excessive amounts of volatile perfluorinated solvents associated with waste streams discharged. The device and mounted sensor may be used to measure one or more fluid properties such as but not limited to temperature, flow, purity, chemical composition, pressure, or a combination of these. The device in a preferred embodiment has a sensor that is coated with a poly-oligomer that includes a fluorine containing aliphatic ether ring structure in the poly-oligomer chain.

[0014] Another embodiment of the present invention is a sensing device for measuring the flow of a fluid comprising: one or more sensors, each sensor having a surface with an effective thickness of an adherent protective coating on a surface of the sensors for contacting a fluid to be measured. The coating including a soluble poly-oligomer containing fluorine, the poly-oligomer chemically bonded to the sensor surface to form an adherent protective coating. The adherent coating is formed by curing a non-porous film of the coating material deposited on the fluid contacting surface of the sensors. The device can include a conduit for directing fluid so that it contacts the coated sensor surface. Structures or means for applying energy, i.e. exciting the sensing device and means or structures for measuring said physical response from the sensors may be present on a surface of the sensors. The device includes a housing that is configured to prevent fluid communication between the non-porous adherent film of the coating material formed on the fluid contacting surface of the sensors and a second fluid isolated surface of the sensors. Each said sensor fluidly sealed to the housing and separated one from the other by a fluid conduit. Preferably the sensors in the device have a film of the coating material formed on the surface of the sensors that includes greater than 10% of the poly-oligomer in the



coating material deposited on the sensors to form the film. The sensors in the device may have an adherent protective coating in their surface which has a surface energy of less than 30 dynes/cm. The device can have sensors that include but are not limited to a material chosen from the group consisting of a piezoresistive, piezoelectric, ceramic, a metal, or a combination of these. The fluid conduit separating said sensors in the device may be a tube and the sensors are pressure sensors. The fluid conduit separating said sensors in the device may be a ventruri and the sensors are pressure sensors. The fluid conduit separating said sensors in the device can be an orifice and the sensors can be pressure sensors. The fluid conduit separating the sensors in the device can be is a tube and the sensors can be pressure sensors. The fluid conduit may be a laminar flow element from a thermal mass flow meter, where the conduit is coated on its fluid contacting inner diameter with the coating material to form the effective thickness of an adherent protective coating on the surface of the sensors for contacting a fluid to be measured; the resistive heating and temperature sensing elements of a thermal flow sensor are wound or deposited on the outside of the tube.

**[0015]** Another embodiment of the present invention is a sensing device that includes a sensor having a surface with an effective thickness of an adherent protective coating. The coating including a soluble poly-oligomer with a fluorine containing aliphatic ether ring structure in the poly-oligomer chain. The poly-oligomer chemically bonds to a sensor surface to form an adherent coating. The adherent coating may be formed by curing a non-porous evaporated film of the coating material formed on or deposited on the surface of the sensor to be coated and in contact with the fluid. Preferably curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-

oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. The non-porous film of the coating material formed on the surface of the sensor in the device preferably includes greater than 10% of the poly-oligomer in the coating material deposited on the substrate to form the non-porous film. The sensing device preferably has a coating on the sensor of effective thickness to protect the underlying sensor from corrosion, erosion, delamination, or particle generation and the adherent protective coating permits the sensor to produce a measurable physical response to a fluid property.

**[0016]** Another embodiment of the present invention is a method of making a coated sensor that includes applying or depositing an amount of a coating material without entraining bubbles in the coating material on a chemically bondable fluid contacting surface of a sensor. The coating material includes a solvent, a soluble poly-oligomer having a fluorine containing groups and an aliphatic ether ring in the chain of the poly-oligomer, and the poly-oligomer has reactive groups. The reactive groups on the poly-oligomer are used for bonding the poly-oligomer to the chemically bondable fluid contacting surface of the sensor. The method has a step of removing solvent from the coating material applied to the chemically bondable fluid contacting surface of the sensor to form a non-porous film of said coating material. The formed non-porous film includes greater than 10% of the poly-oligomer in the coating material originally applied to the fluid contacting surface of the sensor. The method has a step of curing the non-porous film of the coating material on the chemically bondable fluid contacting surface of the sensor. The curing chemically bonds at least a portion the reactive groups of the poly-oligomer to the chemically bondable surface of the sensor surface to form an adherent non-porous protective coating on the chemically bondable fluid contacting surface of the sensor. The method may be used on chemically bondable fluid contacting surface of the sensor that may be

but is not limited to a ceramic, a mineral, a poly-oligomer, a metal or a combination of these. Curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. Preferably the curing step occurs at a temperature below which damage to the electronic structures on other surfaces of the sensor can occur, even more preferably the curing temperature is less than 120°C. Chemical and photochemical treatments of the non-porous film may be used to cure it to the sensor surface. The chemically bondable fluid contacting surface of the sensor used in the method may be treated with an organosilane to form the chemically bondable fluid contacting surface of the sensor. The method may be used to coat one or more sensors, each sensor having a chemically bondable fluid contacting surface. The coating material can be applied to each sensor by a dispenser having one or more nozzles. The chemically bondable fluid contacting surface of the sensor used in the method may include a ceramic material or a ceramic material with a coating on it. The method preferably results in an effective thickness of an adherent non-porous coating on a surface of the sensor which protect it and structures formed on it from corrosion, particle generation, delamination, or changes in sensor response and sensitivity caused by the fluid and even more preferably has a thickness of greater than 50 microns and even more preferably greater than or equal to 100 microns. The method of coating may be used for a sensor which includes a diaphragm. The solvent removal step of the method may be performed in an antistatic environment. The curing step of the method may include but is not limited to chemical, photochemical, thermal curing or a combination of these. The sensor, coated by the method may include structure on one or more of its surfaces chosen from the group

consisting of resistive, capacitive, transistors, electrical contacts, optical contacts, or a combination of these.

**[0017]** Advantage of the present invention include the ability to form thick non-porous protective coatings on a variety of substrates in a single deposition step. The invention permits a variety of sensors, cuvettes, bellows, gas cells, optical window, material handling devices and housings, to be coated and used in hostile and corrosive environments. Preferably the adherent non porous coating protects the underlying structures from corrosion, particle generation, delamination, or changes in sensor response and sensitivity caused by the fluid. In some cases less expensive but more sensitive versions of sensors can be coated with the protective and adherent film and used in corrosive and hostile environment reducing costs and improving performance. The coating process is simple and results in fluid applied to a substrate that can be treated to form non-porous films of the poly-oligomer suitable for curing. The process minimizes waste and chemical consumption and reduces overall cost while maintaining or improving overall performance achieved through the use of low cost sensors. The ability to coat multiple sensors or other fluid contacting structures and to simplify the manufacturing process because devices can be coated having leads, electronics, and bond pads already formed on the structures by the use of a low temperature curing process condition is advantageous.

**[0018]** The defect free and adherent thick coatings made by the present invention provide greater chemical and mechanical resistance than thinner coatings applied to similar structures because diffusion and chemical permeability decrease as coating thickness increases. The ability to make such coatings in a single step makes the use of such coatings possible because of their ease of manufacture and cost effective because multiple sensors may be coated and material waste is minimized.

**[0019]** The coating can also help to preserve the physical integrity of a fluid system by maintaining a fluid tight seal when there is a mechanical failure of the coated substrate. As an example illustrated in FIG. 6, if a gauge type of ceramic pressure sensor is exposed to a pressure condition in excess of its pressure rating, there is a possibility that the brittle sensing diaphragm may fail and allow the process fluid to escape from the fluid system through the sensor's atmospheric vent, potentially causing damage to nearby equipment, or endangering the health of people in the area. If the sensor diaphragm is coated with a defect free, adherent material that is flexible and extensible, fluid system integrity is maintained in case of diaphragm failure. The coating also prevents contamination of the fluid system in the case of failure of the coated component.

## **DESCRIPTION OF THE DRAWINGS**

**[0020]** In part, other aspects, features, benefits and advantages of the embodiments of the present invention will be apparent with regard to the following description, appended claims and accompanying drawings where:

**[0021]** FIG. 1 illustrates a sensor or substrate with a coating material being dispensed without bubbles onto the substrate surface from a nozzle, electrical or optical feed-through connected to the substrate are also shown;

**[0022]** FIG. 2 (A) illustrates a bubble free volume of coating material deposited onto the surface of a substrate, (B) shows that coating conformally covering a sensor substrate of the present invention after evaporation of solvent to form a non-porous film suitable for curing;

**[0023]** FIG. 3 Calibration data for coated sensor with and without the adherent non-porous coating of the present invention at various time intervals;

[0024] FIG. 4 illustrates a process and apparatus for coating multiple substrates on a rotatable stage with a coating material dispensed from a nozzle onto the substrate surfaces.

[0025] FIG. 5 illustrates sensors having non-porous adherent coating configured in a housing for measuring the property of a fluid; the housing can be mounted in a fluid flow circuit and has inlet and outlet ports in the fluid flow path;

[0026] FIG. 6 illustrates a substrate having a non-porous adherent coating on the surface opposite the sensor base.

## **DETAILED DESCRIPTION**

[0027] Before the present compositions and methods are described, it is to be understood that this invention is not limited to the particular molecules, compositions, methodologies or protocols described, as these may vary. It is also to be understood that the terminology used in the description is for the purpose of describing the particular versions or embodiments only, and is not intended to limit the scope of the present invention which will be limited only by the appended claims.

[0028] It must also be noted that as used herein and in the appended claims, the singular forms “a”, “an”, and “the” include plural reference unless the context clearly dictates otherwise. Thus, for example, reference to a “coating poly-oligomer molecule” is a reference to one or more coating poly-oligomer molecules and equivalents thereof known to those skilled in the art, and so forth. Unless defined otherwise, all technical and scientific terms used herein have the same meanings as commonly understood by one of ordinary skill in the art. Although any methods and materials similar or equivalent to those described herein can be used in the practice or testing of embodiments of the present invention, the preferred methods, devices, and materials are now described. All publications mentioned herein are incorporated by reference. Nothing herein is to

be construed as an admission that the invention is not entitled to antedate such disclosure by virtue of prior invention.

[0029] Embodiments of the present invention include adherent coatings of chemically inert high purity poly-oligomers on sensor and other substrates. The coatings are applied and cured on the sensor substrates at relatively low temperatures which permits the coating process to be performed with electronics for conversion of the substrate properties into electrical signal. Structures of the present invention include those having an effective thickness of a protective non-porous coating that is adherent and chemically bonded surface of the structure in contact with a fluid. Structures may include but not limited to a sensor, a conduit, a vessel such as a cuvette or gas sampling cell, bellows, wafer handling equipment, or transparent windows that are to be contacted with a fluid during chemical processing and preferably a fluid whose properties are to be measured or characterized. The protective and adherent coating is chemically inert, has low permeability to fluids, ions and gases. The adherent non-porous coating on a surface of the sensor protect it and structures formed on such as electrodes and electrical structures from corrosion, particle generation, delamination, or changes in sensor response and sensitivity caused by the fluid.

[0030] High purity poly-oligomers containing fluorine or coatings made from them of are preferably those which do not contribute amounts materials considered to be contaminants in the fluid to be contacted with the coated substrate. Preferably the coating materials or the cured coatings contribute less than 1000 ppb v/v of materials considered to be contaminants to the fluid contacted by the coated substrate. Examples of such contaminants may include ions, water or organic solvents, or particulates. Soluble coating materials or formed coatings may analyzed as neat samples or following extraction using mass spectroscopic techniques, elemental analysis,

HPLC, or other techniques. Where it is difficult to obtain high purity soluble coating materials, ion exchange or extraction of deposited coating may be performed to make coatings with acceptable levels of purity.

**[0031]** Bonding pads may be on one or more surfaces of the substrate and used to provide for input and output of electrical and or optical signals from the substrate to processors, amplifiers, through wires or optical fiber. An example of such a sensor is a strain gauge where the resistive elements are applied to the surface of the sensor that is not in contact with the fluid to be measured. In the case of an optical pressure sensor, for example, bonding pads and or electronics are not present on the surface of the substrate. In this case the substrate surface itself or the surface with a reflective coating may be used for measurement.

**[0032]** The pressure sensor of the present invention may be constructed as a piezoresistive or capacitance sensor having a sensing diaphragm. The sensing diaphragm is made of a etched silicon, a ceramic, a metal, or sapphire. The sensor may have a backing plate, sensing diaphragm, silica glass bond between the backing plate and diaphragm, and electrical leads.

**[0033]** A pressure sensor may include a backing plate, a non-porous diaphragm, a sensing element adjacent an inner surface of the diaphragm, and a glass layer of a high strength material that is bonded by glassing to the backing plate and the non-porous diaphragm. The backing plate provides rigidity to the structure. The rigidity of the backing plate resists stresses transmitted from the housing to the sensing elements on the sensor diaphragm. Although the backing plate is not in direct contact with the process medium it is required to be mechanically stable and amenable to high temperature processes. The thermal expansion rate of the backing plate should approximate closely that of the sensing diaphragm. While it is possible to



compensate for thermal effects, a large mismatch will produce stresses during manufacture that may cause the bond between the two pieces to yield over time. Those skilled in the art will appreciate that the non-porous diaphragm may include a Wheatstone bridge or a conductive layer formed thereon as part of a piezoresistive or capacitive type sensor respectively.

**[0034]** To form a piezoresistive sensor a silicon layer is formed on an inner surface of the non-porous diaphragm, wherein a strain gage such as a Wheatstone bridge is formed thereon. The backing plate includes apertures extending therethrough, the apertures being adapted and may contain optical or electrical leads coupled to the sensing element. A change in pressure near the non-porous diaphragm is detectable by the sensing element. An increase and decrease of pressure against the diaphragm causes deflection of the diaphragm which in turn changes the resistances of the strain gage. The changes in resistance is correlated with the pressure adjacent the diaphragm.

**[0035]** Those skilled in the art will appreciate that the backing plate and non-porous diaphragm are constructed of materials having similar thermal expansion rates to avoid unnecessary stress through a wide range of temperatures. As described below in greater detail the pressure sensor may be constructed such that the sensing element may detect an absolute pressure or gage pressure.

**[0036]** The pressure sensor may includes bond pads formed on the diaphragm between the glass layer and the non-porous diaphragm. Without limitation, the preferred embodiment of the bond pads comprise a titanium layer and a diffusion barrier. The doped silicon thin film interconnects the bond pads in a known suitable manner to form the Wheatstone bridge. A window is formed in the glass layer and backing plate, thereby providing access to bond pads. Electrical leads extend through the windows formed in the glass layer and backing plate and the

electrical leads are brazed to the bond pads. The electrical leads are brazed to the bond pads and the glass layer is glassed to the diaphragm and backing plate.

[0037] In an alternate embodiment the diaphragm and sensing element is modified to create a capacitance rather than a piezoresistive sensor. The thin sensing diaphragm, which flexes when pressure is applied, has a capacitive plate formed on the inner surface of the sensing diaphragm and another capacitive plate is formed on the inner surface of the backing plate. One electrical lead is connected to the capacitive plate formed on the inner surface of the sensing diaphragm and another lead is electrically coupled to the inner surface of the backing plate. As the spacing between the diaphragm and the plate vary with pressure the capacitance of the plates changes. This variation in capacitance is detected by an electrically connected sensing element of known suitable construction.

[0038] In yet another alternative embodiment the diaphragm and sensing element are modified to accept an optical fiber. The optical fiber measures the change in shape of the diaphragm as it changes with pressure. This variation in diaphragm shape is detected by light energy from the optical fiber reflected from the fluid isolated side of the diaphragm and related to fluid pressure.

[0039] Methods, means, and structures for exciting sensors and detecting a response are described in the art. Examples of such are disclosed in U.S. Patents: 6,681,787, 'System and method of operation of a digital mass flow controller', Tinsley, et al; 6,640,822, 'System and method of operation of a digital mass flow controller' Tinsley, et al; 6,617,079, 'Process and system for determining acceptibility of a fluid dispense', Pillion, et al; 6,596,148, 'Regeneration of plating baths and system therefore', Belognia, et al; 6,575,027, 'Mass flow sensor interface circuit', Larsen, et al; 6,527,862, 'Flow controller', McLoughlin, et al; 6,449,571, 'System and

method for sensor response linearization', Tarig, et al; 6,445,980, 'System and method for a variable gain proportional-integral (PI) controller', Vyers; the contents of each of these patents are incorporated herein by reference in their entirety.

**[0040]** The coating material is a chemically inert poly-oligomeric material having low surface energy, below about 40 dynes/cm, low chemical permeability to dissolved ions, liquids, and gases, low mass change, incompressible. The poly-oligomer chain has pendant groups that contain fluorine and can be dissolved in a solvent. The protective films preferably are those which can be characterized as having a gas permeability less than Teflon AF® for a test gas such as oxygen or nitrogen. Alternatively non-porous adherent films of the present invention may be characterized for permeation through a sample of the membrane film in contact with a temperature controlled volume of fluid and measuring permeation as a function of temperature, fluid properties, or film thickness, by connecting the outside of the membrane to a detection system like and FTIR spectrometer or an APIMS. Examples of soluble poly-oligomers containing fluorine which may be used as coating material include those disclosed in U.S. Pat. No. 6,201, 085 the contents of which are incorporated herein by reference in their entirety. These coating materials include those which are a perfluoro-poly-oligomer obtained by the cyclo-poly-oligomerization of perfluoro(alkenyl vinyl ether). This poly-oligomer containing fluorine is soluble in organic solvents due to its amorphous structure, and may be coated onto a substrate. The substrate may be any material with which the material can form an adherent film. For example, the substrate may be treated with an adhesion promoter such as an amino-silane coupling agent and then coated with the perfluoro-poly-oligomer. Alternatively the end groups of the perfluoro-poly-oligomer are modified to provide chemical groups that provide adhesion to the substrate as disclosed in U.S. Pat. No. 5,498,657 the contents of which are incorporate herein

by reference in their entirety. For example, the endgroups of the poly-oligomer chain may be modified with an organo-silane which can be used to bond the modified perfluoro-poly-oligomer to the substrate. The use of an adhesion promoter or chemically modified poly-oligomers reactive with the substrate provides adhesion of the poly-oligomer to the substrate and by reacting with substrate surface groups may help to reduce corrosion of the substrate surface. Other useful soluble poly-oligomers containing fluorine pendent groups for coatings may include those disclosed by French et. al, Journal of Fluorine Chemistry, vol 122 (2003) whose end groups can be chemically modified to promote bonding with the substrate surface or may be combined with an adhesion promoter to form adherent films with the substrate.

**[0041]** The use of an adhesion promoter in accordance with the present invention advantageously avoids the disadvantages attendant upon forming separate barrier/passivation layers and separate adhesion layers. In accordance with the present invention, any of various commercially available adhesion promoters can be employed, such as but not limited to silane-based organic adhesion promoters. Suitable commercially available silane-based adhesion promoter include 3-APS (3-aminopropyltriethoxysilane) or MOPS (3-methacryloxypropyltrimethoxysilane). Other commercially available silane-adhesion promoters containing vinyl, chloropropyl, epoxy, diamine, mercapto and/or cationic styryl organofunctional groups can be employed. Where chemically acceptable, non-silicon adhesion promoters containing for example aluminum, gallium, or other elements may be used.

**[0042]** The coating material dissolves in a solvent. The coating material may include fluorine containing poly-oligomers with alicyclic structures in its main chain and preferably fluorine containing aliphatic ether ring structures in its main chain. The chain of the poly-oligomer in the coating material may have reactive groups for bonding to the substrate. The

coating material poly-oligomer may have molecules such as but not limited to amino functionalized organosilanes dissolved in the coating material that may bond to the substrate and to reactive groups on the poly-oligomer chains for bonding the poly-oligomer to the substrate. The coating material poly-oligomer may have reactive groups for bonding to the substrate and for bonding with other poly-oligomer chains.

**[0043]** The molecular weight of the poly-oligomer in the coating material may be used to tailor the coating for surface coverage, adhesion, strength, and chemical permeability. The concentration of the poly-oligomer used in the coating solution will affect its dispense, viscosity, and the thickness of a formed film for a given amount of coating material deposited on a substrate. The concentration of poly-oligomer in the solution depends upon the molecular weight of the poly-oligomer, but may be less than about 50% and is preferably less than about 25% by weight. The density of the the adherent non-porous coating film may be controlled by the concentration of the poly-oligomer in the coating solution as well as by its molecular weight; increasing the poly-oligomer concentration, use of mixtures of fluorine containing poly-oligomers, or increasing the molecular weight may be used to change film density

**[0044]** The coating material includes poly-oligomeric molecules and may be used to refer to both poly-oligomeric molecules and oligomeric molecules; however the present invention may be practiced with oligomeric molecules, or a mixture of oligomers and poly-oligomers. Oligomers which may be used include those fluorine containing molecules with reactive groups similar to those present in the poly-oligomer or poly-oligomeric molecule disclosed herein and especially those reactive groups which may be used to form higher molecular weight poly-oligomeric species upon thermal, chemical, or photochemical curing of an oligomeric coating material.

[0045] The purity of the coating material including but not limited to hydrocarbons, metal ions, and anions is high to prevent unwanted contamination of fluids measured by the sensor of the present invention. It may also be possible to clean deposited films for example by chemical extraction or vacuum baking and degassing to achieve a higher purity in finished films.

[0046] Materials for coating the substrates, and preferably sensor substrates, of the present invention may include those which cure and adhere by chemical bonding to the substrate, form defect free (cracks, voids, bubbles) films of sufficient thickness to provide the substrate with chemical protection and not attenuate the sensitivity of the sensor in its application. The materials may be applied at temperatures and processing conditions which are compatible with sensors having electronic circuitry formed on a portion of the substrate. Curing the non-porous film can occur at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film followed by ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. It is desirable that the coating form a uniform film that adheres to the sensor substrate at temperatures below about 200 °C and more preferably below about 120 °C.

[0047] The temperature for drying the fluorine-containing solvent and fluorine containing poly-oligomer is selected depending upon the heat resistance of the substrate, and may range from 15 °C to about 150 °C; and preferably is below 35 °C. To prevent an irregular film thickness the solvent may be removed in an antistatic environment. Evaporation of solvent from the **coating material composition** forms a non-porous film of the poly-oligomer from the coating material on the substrate. Preferably the film of the coating material on the substrate is non-porous so that fluids or particles cannot pass through the film except by permeation or

diffusion. The coating material film on the substrate, and preferably a non-porous film, may be cured to form the adherent film of the coating material on the surface of the substrate. Long curing times may be used to achieve reaction of the adhesion promoter at lower temperatures. Lower curing temperature permits substrates with deposited electronics to be coated and processed directly which provides for low cost production methods and permits less expensive electronics to be used.

**[0048]** Preferably the adherent non-porous coatings have a low permeability for ions, water, and other fluids so that fluids to be measured are prevented from contacting the substrate and the coating mass and physical properties are maintained. Also, the cured coatings preferably do not attenuate the signal of the sensor and maintain sufficient rigidity.

**[0049]** The coating material may be deposited, applied, or dispensed onto the substrate by low temperature coating processes that preferably minimizes or eliminates waste of the coating material. Examples of such coating method may include but are not limited to knife over roll coating, die cast coating, immersion coating, curtain coating, and air knife coating. Where coating of the substrate utilizes a nozzle, the nozzle can be positioned over the substrate and in a proximity to the substrate so the coating material from the nozzle contacts the surface of the substrate in a continuous stream. One method of avoiding bubble generation is minimizing the velocity of the solution as it is transferred from pipette to diaphragm by careful pipetting or nozzle dispense technique. Dispense pumps such as the Intelligen® from the Mykrolis Corporation, syringe pumps such as the Harvard Model 22, or automated micropipettors can be used to dispense fluid onto substrates. The stream of fluid without the formation of drops also provides for thick coating films and minimize the entrainment of bubbles in the films which may lead to voids and corrosion paths in the coating. Multiple dies may be used for coating multiple

substrates or the substrates may be moved under the die on a rotating table or a conveyer. The coating can be deposited onto the substrate at a rate so that a meniscus of the liquid forms on the sensor .

**[0050]** The coating is preferably applied to the substrate in by a method or process which minimizes and preferably eliminates the amount of coating material which is lost or wasted during the coating process. For example, in the application of coating material onto a planar electrode, the coating material is may be dispensed from a nozzle onto a stationary or slowly rotating electrode in a single continuous stream until the substrate is covered with the coating material. The liquid is allowed to evaporate to leave a non-porous conformal film of the coating on the substrate. By comparison, a spin coating process, while it may be used to distribute dispensed coating material across a substrate, is less desirable because upto about 99% of the coating fluid dispensed onto the coating surface is lost during spin up.

**[0051]** During the dispense and and subsequent evaporation of solvent from the coating material applied to the substrate the composition of the atmosphere surrounding the substrate and coating may be controlled. This may include but is not limited to control of the temperature, flow rate of gas across the coated substrate, composition of the gas to include reactive gases as well as solvent vapor, the use of an antistatic environment or a combination of any of these.

**[0052]** The temperature during evaporation can be in the range of from 15 C to 50 C and provides for a crack free film of the uncured poly-oligomer to form on the sensor substrate. The evaporation parameter to form the non-porous film may be determined by thermogravimetric analysis of the coating material at different temperatures and gas flow conditions. The evaporation time will depend upon the amount of material, the vapor pressure of the solvent(s), surface area, temperature of the substrate and surrounding environment, and flow rate of gases.



These may be modified as would be known to those skilled in the art, however the formed film is free of defects (crack, voids, bubbles).

[0053] Once the poly-oligomer has formed a self supporting film on the substrate it may be cured to promote adhesion of the poly-oligomer to the substrate. For example it is desirable that the coating form a uniform film that adheres to the sensor substrate at temperatures below about 200 °C and more preferably below about 120 °C. The curing temperature may be slowly ramped up to the curing temperature to avoid rapid solvent outgassing and bubble formation. Ramping the temperature up to a value greater than the boiling point of the solvent and greater than the  $T_g$  of the fluorine containing poly-oligomer removes remaining solvent in the film and to cause the terminal groups of the fluorine containing poly-oligomer to further react, either with the ceramic substrate, promoting adhesion, or internally, crosslinking and increasing chemical resistance. This process can be carried out in an oven capable of increasing the process temperature at a rate of about 5 °C/hour, 10 °C/hour or more provided that bubbles or cracks are not formed in the film as temperatures above the solvent boiling point and poly-oligomer glass transition temperature are exceeded. After heating the non-porous film on the substrate to a temperature above the solvent boiling point and poly-oligomer glass transition temperature the substrate is allowed to soak at the final temperature for about 2 hours or sufficient time to chemically bond the film to the substrate. The time required to sufficiently bond the film to the surface of the substrate may vary depending upon the substrate, final temperature, and reactivity of the adhesion promoting groups but can be determined by measuring the non-porous film adhesion with a standard tape test after various temperature and times for heating. An inert gas purge may be used during the evaporation and heat treatment processes.

**[0054]** When water vapor or any other condensable vapor is absorbed by a poly-oligomer film, the physical properties of the film, such as mass, thickness, surface resistance, volume resistance, and dielectric constant, all change. These changes, which can be detected by various means and may be used to characterize the desirability of various coating materials. For instance, changes in the quantity of water absorbed in humidity-sensitive films can be measured as changes in 1) the resonant frequency of a surface acoustic wave or a mechanically resonant structure coated with the film, 2) the surface or volume resistance between two electrodes connected to the film, or 3) the capacitance between sandwich-electrode or interdigitated-electrode capacitors employing the film as dielectric.

**[0055]** A very wide range of substrates may be provided with a composite coating in accordance with the instant invention. Almost any organic or inorganic solid material may be suitable as a substrate, including metal, glass, ceramic semiconductors, rubber, natural and synthetic resins, etc. Especially useful are those substrates which have reactive groups, such as but not limited to hydroxyl and carboxylic acid groups that can be used to chemically react with and bond to a portion of the poly-oligomeric molecules in the coating material or an added adhesion promoter. The process of the invention is also suited for encapsulation of electrode and electronic components. The adherent non-porous films coating a portion of the substrates in contact with a fluid bonds to the surface and reduces or is non-permeable to fluids and ions in fluids which degrade or corrode the underlying substrate. The degradation may affect the physical or chemical properties of the underlying substrate for its intended purpose. The degradation may be used to prevent the release of degradation products, such as but not limited to particles, molecules, and ions, from the underlying substrate into the process fluid

**[0056]** Sensors which may be coated by the materials and methods of the present invention are preferably those on which a layer of poly-oligomer may be applied to form a thick film of the poly-oligomer on the sensor such as but not limited to die cast, aerosol spray coat, dip coating, or a combination of these. The substrates may be coated in a single step, however more than one coating step may be used to achieve a desired film thickness or to coat substrates where it is not practical to coat the entire object in a single step. Preferably the substrates are planar, however the planar substrates may include channels or raised structures on their surface. The sensor substrates may have a surface in which have reactive groups or may be chemically treated such as by plasma, pre-coating, or chemical methods to form groups on the sensor surface that can further react with reactive groups on poly-oligomer molecules in the coating material solution to bond a portion or all of the poly-oligomer to the substrate. The poly-oligomer molecules in addition to bonding with the substrate surface, may also react with each other to form a cross linked structure. The reaction of the reactive groups of the poly-oligomer with each other or with the surface may be initiated by thermal, photochemical, or chemical treatments. For example, the thermal treatment of the substrate and poly-oligomer film can result in hydrolysis reactions between the substrate and reactive groups on the poly-oligomer.

**[0057]** Where the substrate includes one or more material interfaces between different materials a poly-oligomer coating material may be applied to the substrate and one or more interfaces to provide chemical and mechanical advantages to the substrate and interfaces. Preferably each of the surfaces are capable of or have been treated to react with an adhesion promoter or the poly-oligomer molecules in the poly-oligomer coating material to effect adhesion of the coating material to the substrate. Thermal, photochemical, or chemical reaction or a combination of these may be used to react the surfaces with coating to effect adhesion.

**[0058]** For optically transparent poly-oligomer films, a chemically sensitive material may be incorporated into a portion of the poly-oligomer molecules or the chemically sensitive material may be incorporated into the surface of the poly-oligomer film. A change in color or absorption for example may be detected by light absorbed or reflected from the coating to indicate the presence or absence of desirable or deleterious molecules in the fluid. Optical windows may be coated by the fluorine containing poly-oligomer coating material composition to provide chemical and physical protection to the window.

**[0059]** The substrates coated by the poly-oligomer may include but are not limited to pressure sensors including strain gauges, capacitive based pressure sensors, and fiber optic or laser diode based pressure sensors that measure changes in a property, such as intensity, of light or electromagnetic energy reflected from the diaphragm surface. The substrate may serve as a support for the poly-oligomer film and any chemically sensitive material incorporated thereon. In this case the substrate and film are optically transparent in the wavelength region used for detection. The sensor may be used to measure the temperature of a fluid and the poly-oligomeric coating used to prevent or substantially reduce ions and fluid from reaching the temperature probe.

**[0060]** The sensors of the present invention may be mounted in a housing to contact the sensors with a fluid whose property is to be measured. The housing may be made of a chemically suitable material, preferably a material which is chemically inert to the fluid. Some housing materials may include a coating of the poly-oligomeric material applied to a portion or all of the housing surfaces. The poly-oligomer coated sensor or optical window in a housing or vessel may be mounted to or in the housing or vessel using methods and materials known to those skilled in the art including but not limited to compression seals using o-rings, gaskets, and

fusion bonding. The housing may be placed in contact with the fluid by submersion or the housing may have inlet and outlet fluid ports for mounting the sensor in fluid communication with fluid in a conduit. The windows may be those in a gas cell used for the analysis of chemical composition of fluids, the adherent non-porous coating protecting the windows and gas cell from corrosion and particle generation.

[0061] The coated sensors may be combined to form flowmeters and other measuring devices such as but not limited to temperature compensated pressure sensors, flow meters, and flow rate compensated chemical sensors.

[0062] Various aspects of the present invention will be illustrated with reference to the following non-limiting examples.

### **EXAMPLE 1**

[0063] CYTOP coating of the Metallux ceramic pressure sensors to form an integral coating include applying the poly-oligomer solution onto the sensor without entraining air bubbles and slowly ramping up to the curing temperature to avoid rapid solvent outgassing and bubble formation. Examples of Cytop<sup>TM</sup> and its properties are disclosed in Asahi Glass Company Product Information available on the world wide web at URL:[www.agc.co.jp/english/chemicals/shinsei/cytop/cytop.htm](http://www.agc.co.jp/english/chemicals/shinsei/cytop/cytop.htm) dated 1/30/2004 and at URL:[www.bellexinternational.com/Cytop%20Flyer.pdf](http://www.bellexinternational.com/Cytop%20Flyer.pdf) dated 2/20/2004, the contents of each are incorporated herein by reference in their entirety.

[0064] Calibration data from a single Metallux pressure sensor; uncoated, with a 2.5 mil CYTOP coating and with a 5 mil CYTOP coating: there was no significant difference between any of the data sets as illustrated in FIG. 3.

**[0065]** Metallux data show an offset shift < 0.2% F.S. after 2,000,000 F.S. pressure cycles and an offset shift of <0.05% F.S. after 110 hours at 82°C.

## **EXAMPLE 2**

**[0066]** Evaluation of chemical compatibility of Metallux pressure sensors coated with Cytop resin. Extractables data was acquired from tests using HCl, TMAH, and DI water.

**[0067]** The extractables levels from the tests run with DI water were all very low. The results from both the TMAH and HCl showed higher levels of aluminum and the TMAH showed high levels of lead. In both chemicals, the aluminum and lead levels were higher in the sensors coated with a 2.5mil thick layer of Cytop, compared to the sensors coated with a 5mil thick layer of Cytop. The aluminum levels from both chemicals were highest in the aluminum buttons coated with a 5mil thick layer of Cytop.

**[0068]** These tests were run using ten Teflon housings, each of which was designed to hold two Metallux pressure sensors. With the sensors installed, each unit can hold approximately 1.5ml of fluid. When installed, the face of each of the two sensors is in contact with the fluid.

**[0069]** Four different materials were tested: Teflon dummy sensors with the same dimensions as the pressure sensors were used for a baseline, aluminum buttons with the same dimensions as the pressure sensors coated with a 5mil layer of Cytop, Metallux pressure sensors coated with a 5mil layer of Cytop, and Metallux pressure sensors with a 2.5mil layer of Cytop. Three different chemicals were used: deionized water, 10% hydrochloric acid, and 2.5% tetramethyl ammonium hydroxide (TMAH). A description of the material and chemical tested in each unit is shown in Table 1.

**[0070]** Prior to the start of the experiment, the Teflon enclosures, the Kalrez o-rings used to seal the sensors in the units, and the Teflon dummy sensors were pre-extracted over night in 10% HCl. Once the materials and chemicals were installed in each unit, the ten units were placed in a 50 °C oven for one month. Periodically during the month, the units containing the aluminum buttons were tested for resistance. A multimeter was used to test the resistance across the two aluminum buttons in each of these units. The results of each measurement are shown in Table 2. No increase in resistance was observed during any of the measurements indicating that measurable amounts of ions were not generated by diffusion or permeation of ions through the films. At the end of the month, all of the chemical samples were submitted to the Analytical department for extractables testing.

**[0071]** The extractables results are reported in units of ug/unit, where a unit is one housing containing two sensors. The extractables levels from tests run with DI water were all below 1 ug/unit. The highest extractables were seen from the Cytop-coated aluminum button, in which the sodium level was 0.63 ug/unit, the potassium level was 0.59 ug/unit, and the calcium level was 0.24 ug/unit.

**[0072]** The extractables levels from tests run with HCl were all below 1 ug/unit, except for aluminum. The aluminum levels were highest from the Cytop-coated aluminum button (5.41 ug/unit). The aluminum levels from the 2.5mil Cytop-coated sensor (3.51 ug/unit) were much higher than those from the 5mil Cytop-coated sensor (0.15 ug/unit). The aluminum levels from the 5mil Cytop-coated sensor were lower than those from the Teflon button (0.42 ug/unit).

**[0073]** The extractables levels from tests run with TMAH were all below 1 ug/unit, except for aluminum and lead. The aluminum levels were highest from the Cytop-coated aluminum button (6.59 ug/unit). The aluminum levels from the 2.5mil Cytop-coated sensor

(2.18 ug/unit) were five times higher than those from the 5mil Cytop-coated sensor (0.42 ug/unit). The aluminum levels from the 5mil Cytop-coated sensor were close to those from the Teflon button (0.31 ug/unit). The lead levels for very small in the Teflon button and Cytop-coated aluminum button (0.01 ug/unit). The lead levels from the 2.5mil Cytop-coated sensor (2.19 ug/unit) were three times higher than those from the 5mil Cytop-coated sensor (0.69 ug/unit).

Table 1  
Test Unit Description dimensions are in  
mils

| Test Unit | Material            | Coating Thickness (mil) | Coating Defects                      | Chemical |
|-----------|---------------------|-------------------------|--------------------------------------|----------|
| 1         | Aluminum #1         | 5                       | Small dot on outer edge              | DI Water |
|           | Aluminum #2         | 5                       | None                                 |          |
|           |                     |                         |                                      |          |
| 2         | Sensor #C02482/1-8  | 5                       | Small dot on outer edge              | DI Water |
|           | Sensor #C02482/1-12 | 5                       | Small dot on outer edge              |          |
|           |                     |                         |                                      |          |
| 3         | Teflon              | N/A                     | N/A                                  | HCl      |
|           | Teflon              | N/A                     | N/A                                  |          |
|           |                     |                         |                                      |          |
| 4         | Aluminum #6         | 5                       | None                                 | HCl      |
|           | Aluminum #4         | 5                       | None                                 |          |
|           |                     |                         |                                      |          |
| 5         | Sensor #C02482/1-13 | 5                       | Small dot on outer edge              | HCl      |
|           | Sensor #C02482/1-9  | 5                       | Small fibrous imperfection near edge |          |
|           |                     |                         |                                      |          |
| 6         | Sensor #C02482/1-16 | 2.5                     | None                                 | HCl      |
|           | Sensor #C02482/1-15 | 2.5                     | None                                 |          |
|           |                     |                         |                                      |          |
| 7         | Teflon              | N/A                     | N/A                                  | TMAH     |
|           | Teflon              | N/A                     | N/A                                  |          |
|           |                     |                         |                                      |          |
| 8         | Aluminum #3         | 5                       | None                                 | TMAH     |



|    |                     |     |                         |      |
|----|---------------------|-----|-------------------------|------|
|    | Aluminum #5         | 5   | None                    |      |
| 9  | Sensor #C02300/1-10 | 5   | Small dot on outer edge | TMAH |
|    | Sensor #C02300/1-11 | 5   | None                    |      |
| 10 | Sensor #C02482/1-14 | 2.5 | None                    | TMAH |
|    | Sensor #C02482/1-17 | 2.5 | None                    |      |

Table 2  
Aluminum Button Monitoring (Resistance Measurements):

| Date    | HCl  | TMAH | DI Water |
|---------|------|------|----------|
| 2/3/03  | None | N/A  | N/A      |
| 2/4/03  | None | N/A  | N/A      |
| 2/5/03  | None | N/A  | N/A      |
| 2/6/03  | None | N/A  | N/A      |
| 2/7/03  | None | None | None     |
| 2/10/03 | None | None | None     |
| 2/11/03 | None | None | None     |
| 2/13/03 | None | None | None     |
| 2/14/03 | None | None | None     |
| 2/17/03 | None | None | None     |
| 2/20/03 | None | None | None     |
| 2/24/03 | None | None | None     |
| 2/28/03 | None | None | None     |
| 3/5/03  | None | None | None     |
| 3/10/03 | None | None | None     |

[0074] Sensors were extracted for 30 days in 10% HCl. Extraction volume was 1.5mL.

Samples were analyzed for metals using ICPMS.

| SampleID   | 5SENSO07.D | 5SENSO08.D | 5SENSO09.D | 5SENSO10.D |
|------------|------------|------------|------------|------------|
| SampleName | sample #3  | sample #4  | sample #5  | sample #6  |
| SampleType | unit       | unit       | unit       | unit       |

| Comments            | Teflon Button | Aluminum Button (5mil coating) | Sensor (5mil coating) | Sensor (2.5mil coating) |
|---------------------|---------------|--------------------------------|-----------------------|-------------------------|
| <b>Na (ug/unit)</b> | <b>1.06</b>   | <b>0.04</b>                    | <b>0.06</b>           | <b>0.10</b>             |
| Na RSD (%)          | 3.17          | 1.30                           | 3.46                  | 1.58                    |
| Na IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Mg (ug/unit)</b> | <b>0.13</b>   | <b>0.06</b>                    | <b>0.01</b>           | <b>0.03</b>             |
| Mg RSD (%)          | 2.69          | 2.69                           | 2.75                  | 1.93                    |
| Mg IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Al (ug/unit)</b> | <b>0.42</b>   | <b>5.41</b>                    | <b>0.15</b>           | <b>3.51</b>             |
| Al RSD (%)          | 2.85          | 3.38                           | 3.12                  | 4.03                    |
| Al IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>K (ug/unit)</b>  | <b>0.94</b>   | <b>0.03</b>                    | <b>0.04</b>           | <b>0.07</b>             |
| K RSD (%)           | 4.84          | 2.57                           | 3.41                  | 2.36                    |
| K IDL (ug/unit)     | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Ca (ug/unit)</b> | <b>0.39</b>   | <b>0.06</b>                    | <b>0.07</b>           | <b>0.08</b>             |
| Ca RSD (%)          | 2.55          | 2.36                           | 3.18                  | 2.05                    |
| Ca IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Ti (ug/unit)</b> | <b>0.08</b>   | <b>0.01</b>                    | <b>0.01</b>           | <b>0.01</b>             |
| Ti RSD (%)          | 3.78          | 1.52                           | 2.70                  | 3.34                    |
| Ti IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Cr (ug/unit)</b> | <b>0.04</b>   | <b>0.01</b>                    | <b>0.00</b>           | <b>0.01</b>             |
| Cr RSD (%)          | 3.38          | 4.23                           | 5.24                  | 3.82                    |
| Cr IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Mn (ug/unit)</b> | <b>0.01</b>   | <b>0.01</b>                    | <b>0.01</b>           | <b>0.00</b>             |
| Mn RSD (%)          | 2.81          | 2.79                           | 5.27                  | 2.87                    |
| Mn IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Fe (ug/unit)</b> | <b>0.45</b>   | <b>0.05</b>                    | <b>0.04</b>           | <b>0.04</b>             |
| Fe RSD (%)          | 3.86          | 2.39                           | 3.34                  | 2.53                    |
| Fe IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Ni (ug/unit)</b> | <b>0.02</b>   | <b>0.00</b>                    | <b>0.00</b>           | <b>0.00</b>             |
| Ni RSD (%)          | 2.20          | 6.71                           | 2.46                  | 3.65                    |
| Ni IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Cu (ug/unit)</b> | <b>0.12</b>   | <b>0.02</b>                    | <b>0.02</b>           | <b>0.01</b>             |
| Cu RSD (%)          | 2.90          | 4.19                           | 3.46                  | 3.20                    |
| Cu IDL              | 0.00          | 0.00                           | 0.00                  | 0.00                    |

|                     |             |             |             |             |
|---------------------|-------------|-------------|-------------|-------------|
| (ug/unit)           |             |             |             |             |
| <b>Zn (ug/unit)</b> | <b>0.14</b> | <b>0.00</b> | <b>0.00</b> | <b>0.01</b> |
| Zn RSD (%)          | 2.18        | 2.79        | 2.31        | 1.29        |
| Zn IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Pb (ug/unit)</b> | <b>0.01</b> | <b>0.00</b> | <b>0.04</b> | <b>0.35</b> |
| Pb RSD (%)          | 1.60        | 0.75        | 1.57        | 1.64        |
| Pb IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |

[0075] Sensors were extracted for 30 days in TMAH. Extraction volume was 1.5mL.

TMAH extractions were prepared for analysis by hotplate evaporation and reconstitution in HNO<sub>3</sub>. Samples were analyzed for metals using ICPMS.

| SampleID            | 5SENSO03.D    | 5SENSO04.D                     | 5SENSO05.D            | 5SENSO06.D              |
|---------------------|---------------|--------------------------------|-----------------------|-------------------------|
| SampleName          | sample #7     | sample #8                      | sample #9             | sample #10              |
| SampleType          | unit          | unit                           | unit                  | unit                    |
| Comments            | Teflon Button | Aluminum Button (5mil coating) | Sensor (5mil coating) | Sensor (2.5mil coating) |
| <b>Na (ug/unit)</b> | <b>0.08</b>   | <b>0.04</b>                    | <b>0.07</b>           | <b>0.08</b>             |
| Na RSD (%)          | 1.84          | 6.40                           | 0.65                  | 1.62                    |
| Na IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Mg (ug/unit)</b> | <b>0.02</b>   | <b>0.02</b>                    | <b>0.00</b>           | <b>0.00</b>             |
| Mg RSD (%)          | 1.83          | 5.02                           | 2.47                  | 1.44                    |
| Mg IDL (ug/unit)    | 0.00          | 0.00                           | 0.00                  | 0.00                    |
| <b>Al (ug/unit)</b> | <b>0.31</b>   | <b>6.59</b>                    | <b>0.42</b>           | <b>2.18</b>             |
| Al RSD (%)          | 3.65          | 6.21                           | 4.43                  | 2.69                    |
| Al IDL              | 0.00          | 0.00                           | 0.00                  | 0.00                    |

|                     |             |             |             |             |
|---------------------|-------------|-------------|-------------|-------------|
| (ug/unit)           |             |             |             |             |
| <b>K (ug/unit)</b>  | <b>0.05</b> | <b>0.02</b> | <b>0.03</b> | <b>0.02</b> |
| K RSD (%)           | 2.01        | 5.33        | 0.28        | 1.07        |
| K IDL (ug/unit)     | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Ca (ug/unit)</b> | <b>0.06</b> | <b>0.01</b> | <b>0.01</b> | <b>0.01</b> |
| Ca RSD (%)          | 2.14        | 5.01        | 1.00        | 1.16        |
| Ca IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Ti (ug/unit)</b> | <b>0.01</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> |
| Ti RSD (%)          | 8.58        | 8.19        | 3.62        | 4.37        |
| Ti IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Cr (ug/unit)</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> |
| Cr RSD (%)          | 2.78        | 5.55        | 2.54        | 2.33        |
| Cr IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Mn (ug/unit)</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> |
| Mn RSD (%)          | 4.15        | 6.18        | 4.81        | 4.06        |
| Mn IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Fe (ug/unit)</b> | <b>0.04</b> | <b>0.01</b> | <b>0.01</b> | <b>0.02</b> |
| Fe RSD (%)          | 3.07        | 5.31        | 2.23        | 3.03        |
| Fe IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Ni (ug/unit)</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> | <b>0.00</b> |
| Ni RSD (%)          | 2.54        | 2.68        | 1.18        | 2.68        |
| Ni IDL (ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Cu (ug/unit)</b> | <b>0.01</b> | <b>0.03</b> | <b>0.01</b> | <b>0.00</b> |
| Cu RSD (%)          | 2.38        | 4.54        | 1.06        | 2.10        |
| Cu IDL              | 0.00        | 0.00        | 0.00        | 0.00        |

|                        |             |             |             |             |
|------------------------|-------------|-------------|-------------|-------------|
| (ug/unit)              |             |             |             |             |
| <b>Zn</b><br>(ug/unit) | <b>0.01</b> | <b>0.13</b> | <b>0.02</b> | <b>0.01</b> |
| Zn RSD<br>(%)          | 6.79        | 1.34        | 2.32        | 4.60        |
| Zn IDL<br>(ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |
| <b>Pb</b><br>(ug/unit) | <b>0.01</b> | <b>0.01</b> | <b>0.69</b> | <b>2.19</b> |
| Pb RSD<br>(%)          | 18.86       | 4.06        | 5.20        | 4.28        |
| Pb IDL<br>(ug/unit)    | 0.00        | 0.00        | 0.00        | 0.00        |

[0076]

[0077] Sensors were extracted for 30 days in water. Extraction volume was 1.5mL.

Samples were analyzed for metals using ICPMS.

| SampleID               | 5SENSO01.D                     | 5SENSO02.D            |
|------------------------|--------------------------------|-----------------------|
| SampleName             | sample #1                      | sample #2             |
| SampleType             | unit                           | unit                  |
| Comments               | Aluminum Button (5mil coating) | Sensor (5mil coating) |
| <b>Na</b><br>(ug/unit) | <b>0.63</b>                    | <b>0.12</b>           |
| Na RSD<br>(%)          | 2.24                           | 6.61                  |
| Na IDL<br>(ug/unit)    | 0.00                           | 0.00                  |
| <b>Mg</b><br>(ug/unit) | <b>0.04</b>                    | <b>0.02</b>           |
| Mg RSD<br>(%)          | 3.16                           | 9.34                  |
| Mg IDL<br>(ug/unit)    | 0.00                           | 0.00                  |
| <b>Al</b><br>(ug/unit) | <b>0.08</b>                    | <b>0.18</b>           |
| Al RSD<br>(%)          | 2.91                           | 6.21                  |
| Al IDL<br>(ug/unit)    | 0.00                           | 0.00                  |
| <b>K (ug/unit)</b>     | <b>0.59</b>                    | <b>0.13</b>           |
| K RSD (%)              | 2.73                           | 6.76                  |

|                        |             |             |
|------------------------|-------------|-------------|
| K IDL<br>(ug/unit)     | 0.00        | 0.00        |
| <b>Ca</b><br>(ug/unit) | <b>0.24</b> | <b>0.09</b> |
| Ca RSD<br>(%)          | 3.51        | 12.34       |
| Ca IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Ti</b><br>(ug/unit) | <b>0.00</b> | <b>0.00</b> |
| Ti RSD<br>(%)          | 3.87        | 2.92        |
| Ti IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Cr</b><br>(ug/unit) | <b>0.00</b> | <b>0.00</b> |
| Cr RSD<br>(%)          | 2.37        | 7.72        |
| Cr IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Mn</b><br>(ug/unit) | <b>0.00</b> | <b>0.00</b> |
| Mn RSD<br>(%)          | 3.86        | 14.44       |
| Mn IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Fe</b><br>(ug/unit) | <b>0.01</b> | <b>0.01</b> |
| Fe RSD<br>(%)          | 3.07        | 10.80       |
| Fe IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Ni</b><br>(ug/unit) | <b>0.00</b> | <b>0.00</b> |
| Ni RSD<br>(%)          | 3.47        | 10.98       |
| Ni IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Cu</b><br>(ug/unit) | <b>0.00</b> | <b>0.01</b> |
| Cu RSD<br>(%)          | 7.31        | 34.29       |
| Cu IDL<br>(ug/unit)    | 0.00        | 0.00        |
| <b>Zn</b><br>(ug/unit) | <b>0.01</b> | <b>0.01</b> |

|                     |             |             |
|---------------------|-------------|-------------|
| Zn RSD (%)          | 3.64        | 1.74        |
| Zn IDL (ug/unit)    | 0.00        | 0.00        |
| <b>Pb (ug/unit)</b> | <b>0.00</b> | <b>0.04</b> |
| Pb RSD (%)          | 3.48        | 3.82        |
| Pb IDL (ug/unit)    | 0.00        | 0.00        |

### EXAMPLE 3

**[0078]** Using volumes of Cytop coating material greater than about 200  $\mu$ l, the coating material solution will spread across a sensor surface that is an octagon shape with an edge to edge length across the sensor of about 0.654 inches and area 0.35 square inches.

**[0079]** To avoid generation of small bubbles in the Cytop coating applied to the sensor surface, these bubbles expand upon heating and create void or porous films, it is preferably not to blow out the pipette tip – this always produces bubbles. Hold tip just above sensor/coating surface to let solution drops spread w/o producing bubbles and better still without producing individual drop. Let solvent evaporate for at least 30 min. before heating in oven. A longer evaporation. time might be necessary for the second and subsequent coats

### EXAMPLE 4

**[0080]** This example describes COATING METALLUX 501 CERAMIC SENSORS WITH CYTOP PERFLUOROPOLY-OLIGOMER, available from Asahi Glass Company. CYTOP™ is a perfluoro-polymer obtained by the cyclopolymerization of perfluoro(alkenyl vinyl ether and contains fluorine bonded to the carbon chain.

**[0081]** The purpose of coating the wetted surfaces of a ceramic pressure sensor with CYTOP perfluoropoly-oligomer is to enhance the chemical resistance of these surfaces thereby minimizing the risk of contaminating the process fluid coming into contact with the sensor.

**[0082]** The coating process in this example includes the steps of: cleaning and dehydrating the surface(s) to be coated, applying a bubble-free coating of CYTOP P(er)F(luoro)P(olymer) solution to the sensor surface, allowing the bulk of the solvent to evaporate at room temperature, soft baking the sensor at a temperature less than the boiling point of the coating material solvent and less than the Tg of CYTOP PFP to remove most of the remaining solvent, ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the CYTOP material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate. For coatings thicker than can be applied to the surface in one coat, the steps of applying, evaporating, and curing can be repeated as needed to achieve the desired thickness prior to the final bake or curing cycle.

**[0083]** Cleaning and dehydrating the surface to be coated can be performed to ensure good adhesion between the sensor and the CYTOP coating, the surfaces to be coated (sensor diaphragm) should be scrubbed with an acetone wet wipe to remove any organic material and then flushed with a squirt (~1 ml.) of acetone to remove any remaining particulate material. The sensor should then be baked in a 110°C oven for 60 minutes to drive off any moisture. At the end of the bake cycle, the hot sensor can be placed in a dessicator to cool, and remain in the dessicator until coated.

**[0084]** Applying a bubble-free coating of CYTOP PFP (PerFluoroPoly-oligomer) solution to the diaphragm. The coating material is 'CTL-107M', a 7% (by weight) solution of



Asahi's 'M' CYTOP perfluoropoly-oligomer (low molecular weight grade) in CT-SOLV100, a perfluoroalkane (primarily perfluoro-octane).

**[0085]** The coating process requires that the sensor be mounted, diaphragm up, in a fixture having an 18 mm. Diameter recess to locate the device. A 15.25 mm. diameter thru hole in the center of the recess provides clearance for the electrical connectors/cables. The recess is level to provide a uniform coating thickness. If the sensor has an electrical cable, the center of mass of the cable falls within the vertical extension of the sensor's O.D. to prevent tilting of the sensor in the fixture.

**[0086]** Once the sensor is properly fixtured, a volume of CYTOP solution sufficient to cover the entire diaphragm surface, but not enough to overcome the surface tension forces at the edges of the diaphragm and allow the solution to spill over the edges, is applied to the diaphragm using a precision microliter pipetter. It was experimentally determined that at least 200  $\mu$ l. of solution was needed to completely cover the diaphragm surface. (The use of more dilute CYTOP 107M solutions resulted in coating thickness variations on the active surface [minimal thermal mass] area of the diaphragm, apparently due to evaporative cooling effects).

**[0087]** Exclude or immediately remove any bubbles from the solution on the diaphragm as they will form defects (pinholes or thin spots) in the coating. One method of avoiding bubble generation is minimizing the velocity of the solution as it is transferred from pipette to diaphragm by careful pipetting technique. Also, the 1 ml. pipetter tips used in the coating process have a very small opening at the tip which can result in high solution dispense velocities and the entrainment of air bubbles in the coating. By cutting back the tapered pipetter tip by about 5 mm., the diameter of the tip opening is increased enough to minimize the generation of bubbles due to high dispense velocities. Visible bubbles in the liquid coating may be moved to the edge

of the diaphragm with the pipetter tip or sucked into the pipetter; however these actions may result in the formation of small, visually undetectable bubbles which eventually become defects in the coating.

**[0088]** Allowing the bulk of the solvent to evaporate at R.T. After the bubble-free coating of CYTOP PFP solution has been applied to the sensor diaphragm, the bulk of the CT SOLV-100 is allowed to evaporate at R.T. for approximately 30 minutes before moving the sensor. This will result in a sensor with a physically stable coating precursor which can be safely transferred to the 'softbake' or curing oven.

**[0089]** 'Soft' baking the sensor at a temperature less than the boiling point of the solvent and less than the T<sub>g</sub> of CYTOP to remove most of the solvent. The boiling point of the CT-SOLV100 solvent is about 100 °C and the T<sub>g</sub> of the CYTOP poly-oligomer is 108 °C. The purpose of this process step is to remove most of the solvent in the coating without initiating further reaction of the poly-oligomer's end groups in preparation for additional coatings or the final bake step. This step is preferentially carried out with the sensor in the same (or a similar) fixture as used in the coating process to keep the sensor essentially level while ramping up from a temperature of 40 °C to a temperature of 60 °C over a period of 60 minutes.

**[0090]** The three previous coating, evaporation and soft bake' steps may be repeated as needed to achieve the desired final coating thickness

**[0091]** Ramping the temperature up to a value greater than the boiling point of the solvent and greater than the T<sub>g</sub> of CYTOP removes remaining solvent in the film and enhances the adhesion of the coating film to the substrate. The purpose of this step is to remove any remaining solvent from the diaphragm's coating and to cause the terminal groups of the CYTOP 'M' poly-oligomer to further react, either with the ceramic substrate, promoting adhesion, or internally, crosslinking

and increasing chemical resistace. This process is carried out in an oven capable of increasing the process temperature at a rate of about 5 °C/hour from 60 °C to 125 °C, followed by a 2 hour 'soak' at 125 °C.

**[0092]** Although the present invention has been described in considerable detail with reference to certain preferred embodiments thereof, other versions are possible. Therefore the spirit and scope of the appended claims should not be limited to the description and the preferred versions contain within this specification.

## CLAIMS

What is claimed is:

1. A device comprising:

a sensor including an effective thickness of a non-porous adherent protective coating on a surface for contacting a fluid to be measured, said coating including a soluble poly-oligomer containing fluorine, and being chemically bonded to said surface.

2. The device of claim 1 wherein said sensor include structure for probing said sensor and structures for measuring said physical response of said sensor.

3. The device of claim 1 wherein said sensor includes a ceramic material.

4. The device of claim 1 wherein said non-porous film of the coating material formed on the surface of the sensor includes greater than 10% of the poly-oligomer in the coating material deposited on the substrate to form the non-porous film.

5. The device of claim 1 wherein said sensor is used measure one or more fluid properties chosen from the group consisting of temperature, flow, chemical purity, pressure, or a combination of these.

6. The device of claim 1 wherein said sensor includes structure on one or more of its surfaces chosen from the group consisting of resistive, capacitive, transistors, electrical contacts, optical contacts, or a combination of these.

7. The device wherein the poly-oligomer coating material includes an organosilane adhesion promoter.

8. The device of claim 1 wherein said adherent protective coating is on one or more surfaces of the sensor.

9. The device of claim 1 wherein said adherent protective coating is at least 50 micron thick.

10. The device of claim 1 wherein the sensor surface is treated to chemically bond said poly-oligomer to the sensor surface.
11. The device of claim 1 wherein said poly-oligomer includes reactive groups for bonding the poly-oligomer containing fluorine to the sensor surface.
12. A method for measuring property of a fluid comprising:
  - contacting said fluid with a sensor having a surface with an effective thickness of an adherent protective coating on the surface of the sensor for contacting the fluid to be measured, said coating including a soluble poly-oligomer containing fluorine, said poly-oligomer chemically bonded to said surface to form an adherent protective coating, said coating formed by curing a non-porous film of the coating material formed on the surface of the sensor.
  - applying energy to said sensing device, and
  - measuring a physical response and comparing said physical response to a standard relationship correlating said physical response to a property.
13. The method of claim 12 wherein said sensor is a piezoresistive, piezoelectric, or thermoresistive material.
14. The method of claim 12 wherein said sensor includes one or more plate in capacitive sensor, having two plates and the poly-oligomeric fluorine containing coating is positioned on one of said plates on a surface in contact the fluid.
15. A method of making a coated sensor comprising:
  - providing a chemically bondable fluid contacting surface on the sensor
  - depositing a non-porous film of a coating material on said surface, said non-porous film of coating material including a soluble poly-oligomer containing fluorine and reactive groups; and
  - curing said deposited non-porous film of said coating material.

16. The method of claim 15 wherein said non-porous film of the coating material on the chemically bondable fluid contacting surface of the sensor is formed by removing solvent from a solution of the poly-oligomer contacting surface of the sensor.
17. The method of claim 15 wherein said curing step occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the surface.
19. The method of claim 15 wherein said fluorine containing poly-oligomer includes an aliphatic ether ring in the chain of the poly-oligomer.
20. The method of claim 15 wherein the fluid contacting surface is made chemically bondable by a treatment chosen from the group consisting of plasma etching, chemical modification with an adhesion promoter, and hydroxylation of a surface .
21. The method of claim 15 wherein said sensor includes structure on one or more of its surfaces chosen from the group consisting of resistive, capacitive, transistors, electrical contacts, optical contacts, and a combination of these..
22. The method of claim 15 wherein said coating material includes an organosilane adhesion promoter.
23. The method of claim 15 wherein said adherent protective coating on the surface of the sensor has a thickness of greater than 50 microns.
24. The method of claim 15 wherein said sensor is a diaphragm.
25. The method of claim 15 wherein sensing means are formed on said sensor prior to coating.
26. The method of claim 16 wherein said solvent removal is in an antistatic environment.
27. The method of claim 15 wherein said curing is thermal curing.
28. A sensing device for measuring property of a fluid comprising:

a sensor having a surface with an effective thickness of an adherent protective coating on a surface of the sensor for contacting a fluid to be measured, said coating including a soluble poly-oligomer containing fluorine, said poly-oligomer chemically bonded to said surface to form an adherent protective coating;

a fluid conduit for directing fluid so it contact the sensor surface; structures for measuring a physical response from said contact; and

a housing to prevent fluid communication between said fluid and a second surface of the sensor.

29. The device of claim 28 wherein said adherent protective coating has a surface energy of less than 30 dynes/cm.

30. The device of claim 28 wherein said housing include fluid inlet and fluid outlet ports.

31. The device of claim 28 wherein said sensor includes a piezoresistive, piezoelectric, or thermoresistive material or a combination of these.

32. The device of claim 28 wherein the non-porous coating of the coating material formed on the surface of the sensor includes greater than 10% of the poly-oligomer in the coating material deposited on the sensors to form the film.

33. The device of claim 28 wherein said sensor measures one or more fluid properties chosen from the group consisting of temperature, flow, purity, pressure, or a combination of these.

34. The device of claim 28 wherein said poly-oligomer includes a fluorine containing aliphatic ether ring structure in the poly-oligomer chain.

35. A sensing device for measuring the flow of a fluid comprising:

one or more sensors, each sensor having a surface with an effective thickness of a non-porous adherent protective coating on a surface of the sensors for contacting a fluid to be measured, said coating including a soluble poly-oligomer containing

fluorine, said poly-oligomer chemically bonded to said surface to form the adherent protective coating, said coating formed by curing a non-porous film of the coating material deposited on the fluid contacting surface of the sensors;

means for applying energy to said sensing device and means for measuring said physical response; and

a housing preventing fluid communication between the non-porous film of the coating material formed on the fluid contacting surface of the sensors and a second fluid isolated surface of the sensors, each said sensor fluidly sealed to the housing and separated one from the other by a fluid conduit.

36. The device of claim 35 wherein the non-porous film of the coating material formed on the surface of the sensors includes greater than 10% of the poly-oligomer in the coating material deposited on the sensors to form the film.
37. The device of claim 35 wherein said adherent protective coating has a surface energy of less than 30 dynes/cm.
38. The device of claim 35 wherein said sensor includes a material chosen from the group consisting of a piezoresistive material, a ceramic material, a metal, or a combination of these.
39. The device of claim 35 wherein said fluid conduit separating said sensors is a tube and the sensors are pressure sensors.
40. The device of claim 35 wherein said fluid conduit separating said sensors is a ventruri and the sensors are pressure sensors.
41. The device of claim 35 wherein said fluid conduit separating said sensors is an orifice and the sensors are pressure sensors.
42. The device of claim 35 wherein said fluid conduit separating said sensors is a tube and the sensors are pressure sensors



43. The device of claim 35 wherein said fluid conduit is a laminar flow element from a thermal mass flow meter, said conduit coated on its fluid contacting inner diameter with the coating material to form the effective thickness of an adherent protective coating on the surface of the sensors for contacting a fluid to be measured.
44. A sensing device comprising:
- a sensor having a surface with an effective thickness of a non-porous adherent protective coating, said coating including a soluble poly-oligomer with a fluorine containing aliphatic ether ring structure in the poly-oligomer chain, said poly-oligomer chemically bonded to said surface to form an adherent coating.
45. The sensing device of claim 44 wherein said non-porous coating material formed on the surface of the sensor includes greater than 10% of the poly-oligomer in the coating material deposited on the substrate to form the non-porous coating.
46. The sensing device of claim 44 wherein the effective thickness of the non-porous adherent protective coating permits said sensor to produce a measurable physical response.
47. A method of making a coated sensor comprising:
- applying an amount of a coating material without entraining bubbles in the coating material on a chemically bondable fluid contacting surface of a sensor, said coating material including a solvent, a soluble poly-oligomer having a fluorine containing an aliphatic ether ring in the chain of the poly-oligomer, and reactive groups on the poly-oligomer, said reactive groups bonding said poly-oligomer to the chemically bondable fluid contacting surface of the sensor;
  - removing solvent from the coating material applied to the chemically bondable fluid contacting surface of the sensor to form a non-porous film of said coating material, wherein the non-porous film includes greater than 10% of the poly-oligomer in the coating material applied to the fluid contacting surface of the sensor; and

- curing said non-porous film of said coating material on the chemically bondable fluid contacting surface of the sensor, said curing chemically bonding at least a portion the reactive groups of the poly-oligomer to the chemically bondable surface of the sensor surface to form an adherent non-porous protective coating on the chemically bondable fluid contacting surface of the sensor.
48. The method of claim 47 wherein the chemically bondable fluid contacting surface of the sensor is a ceramic, a mineral, a poly-oligomer, a metal or a combination of these.
49. The method of claim 47 wherein said curing occurs at a temperature less than the boiling point of the coating material solvent and less than the Tg of poly-oligomer film and ramping the temperature up to a value greater than the boiling point of the solvent and greater than the Tg of the poly-oligomer material to remove any remaining solvent and to enhance the adhesion of the coating to the ceramic substrate..
50. The method of claim 47 wherein said chemically bondable fluid contacting surface of the sensor is treated with an organosilane to form the chemically bondable fluid contacting surface of the sensor.
51. The method of claim 47 wherein one or more sensors, each sensor having a chemically bondable fluid contacting surface, have coating material applied to each sensor by a dispenser having one or more nozzles.
52. The method of claim 47 wherein wherein the chemically bondable fluid contacting surface of the sensor includes a ceramic material.
53. The method of claim 47 wherein said coating has a thickness of greater than 50 microns.
54. The method of claim 47 wherein said sensor includes a diaphragm.
55. The method of claim 47 wherein said solvent removal is in an antistatic environment.
56. The method of claim 47 wherein said curing step is thermal curing.

57. The method of claim 47 wherein the sensor includes structure on one or more of its surfaces chosen from the group consisting of resistive, capacitive, transistors, electrical contacts, optical contacts, or a combination of these.

## **ABSTRACT OF THE DISCLOSURE**

The present invention include non-porous adherent coatings of chemically inert high purity poly-oligomers deposited on substrates. The coatings are applied and cured on the substrates at relatively low temperatures which permits the coating process to be performed with temperature sensitive structures such as electronic circuits, electrodes, and bonding pads in place on the substrate. Coated substrates, such as sensors and fluid conduits, have an effective thickness of the protective non-porous coating that is chemically bonded to a surface of the substrate that will be contacted with a fluid. The adherent non-porous coating on the substrate protect it from corrosion, particle generation, or delamination of structures on the substrate caused by contact with the fluid.

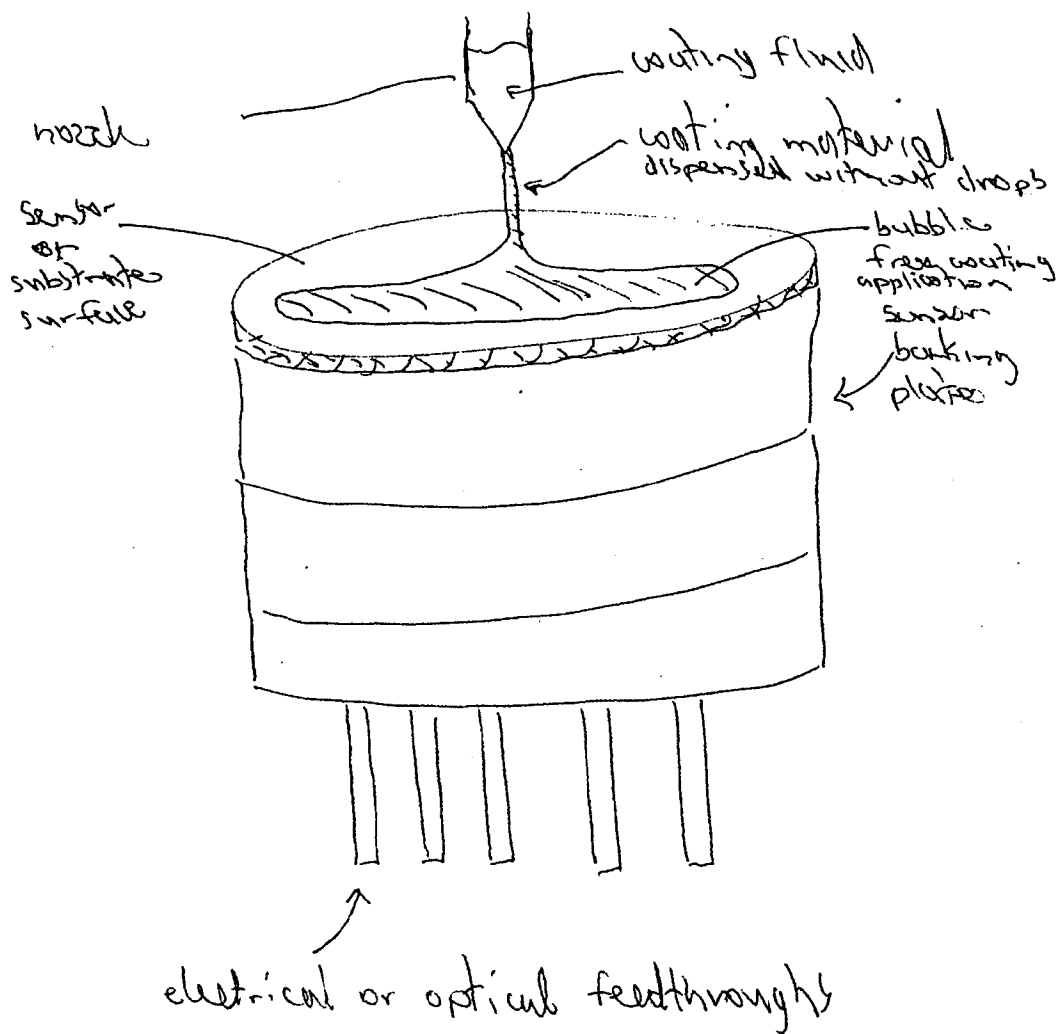
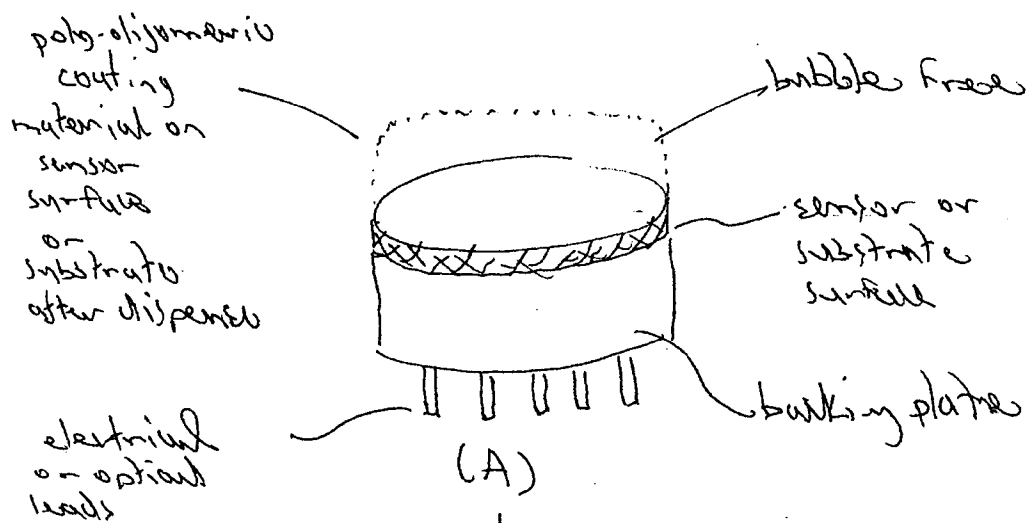


FIG. 1



↓ evaporation/solvent removal @ RT or below  
 ↓ 55°C

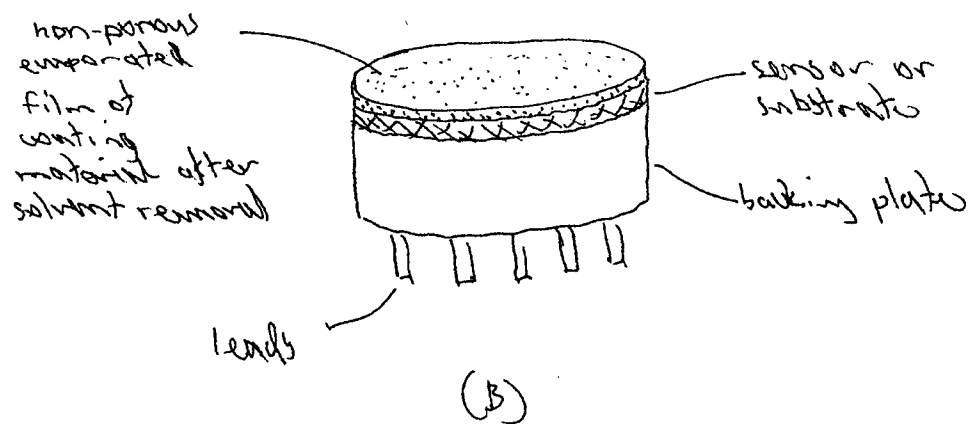
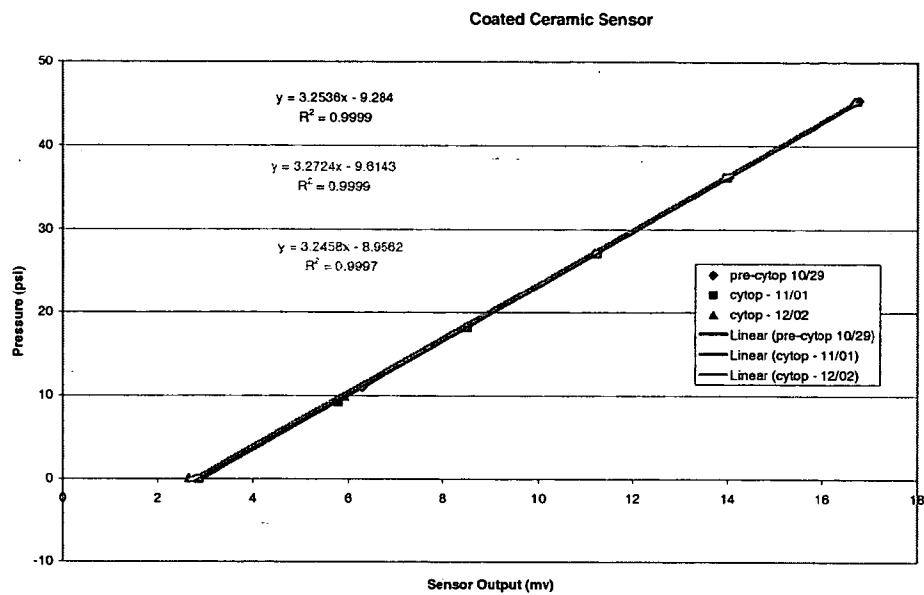


FIG. 2



**FIG. 3**

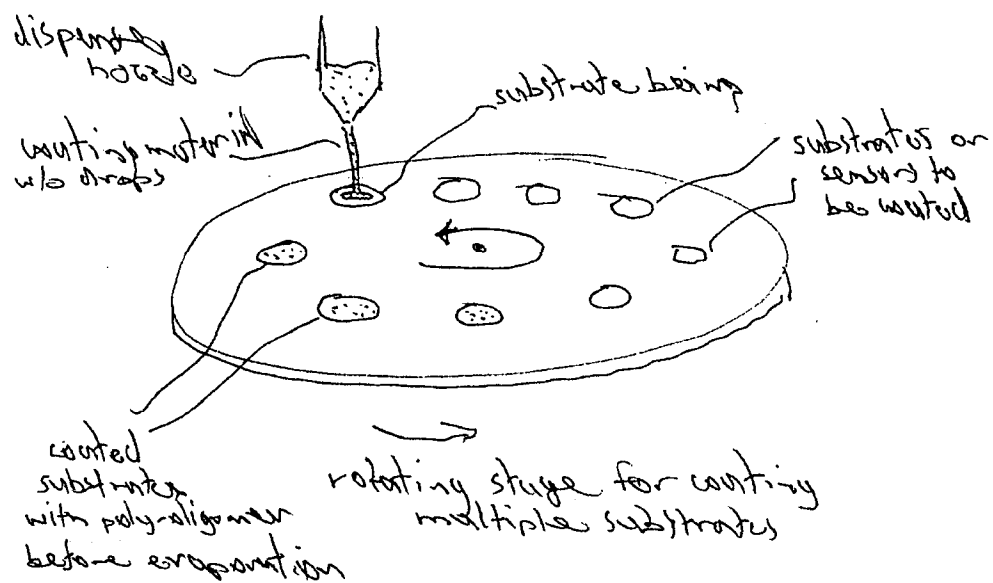
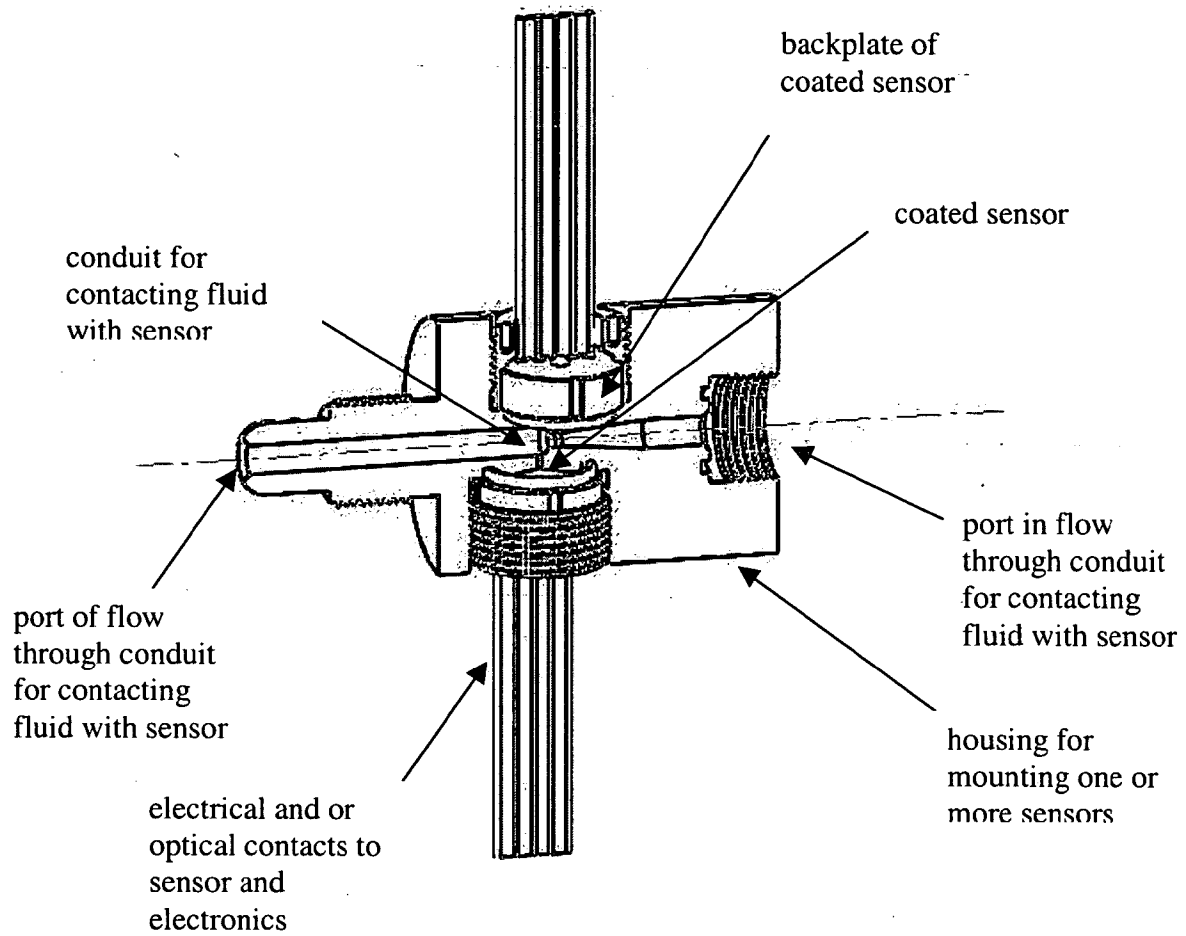


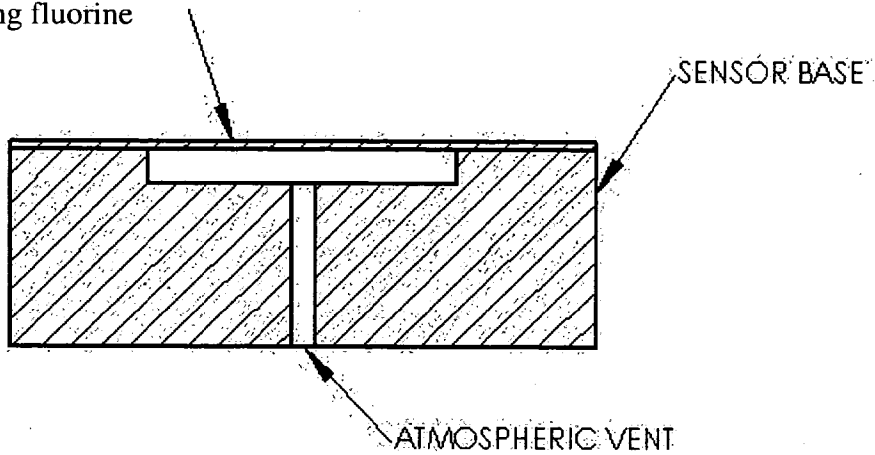
FIG. 4



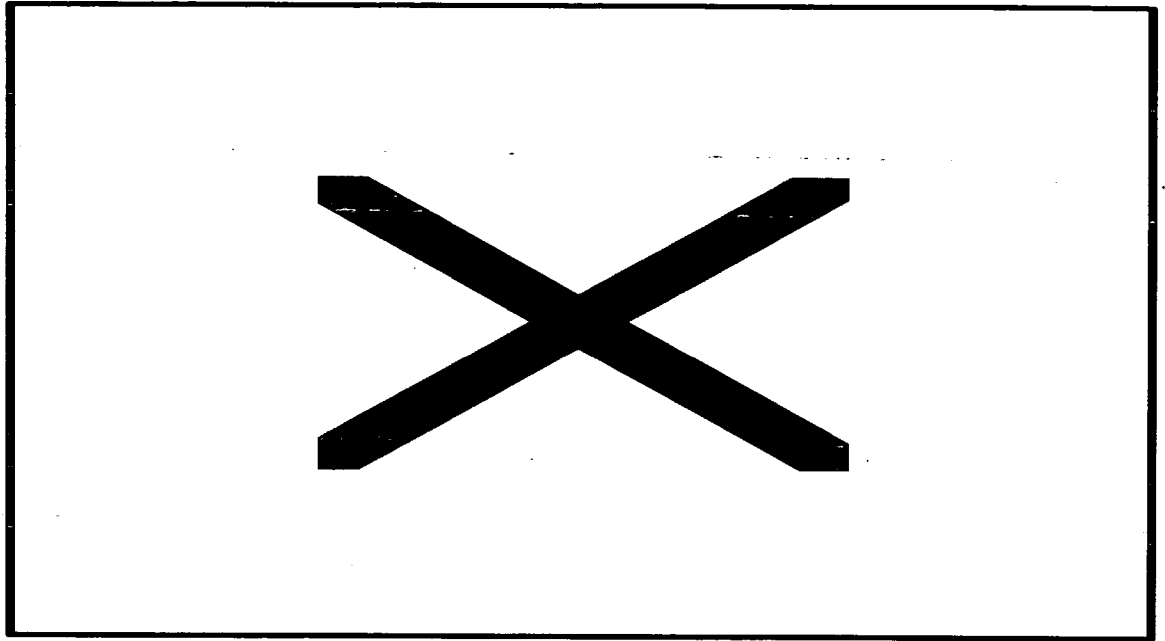


**FIG. 5**

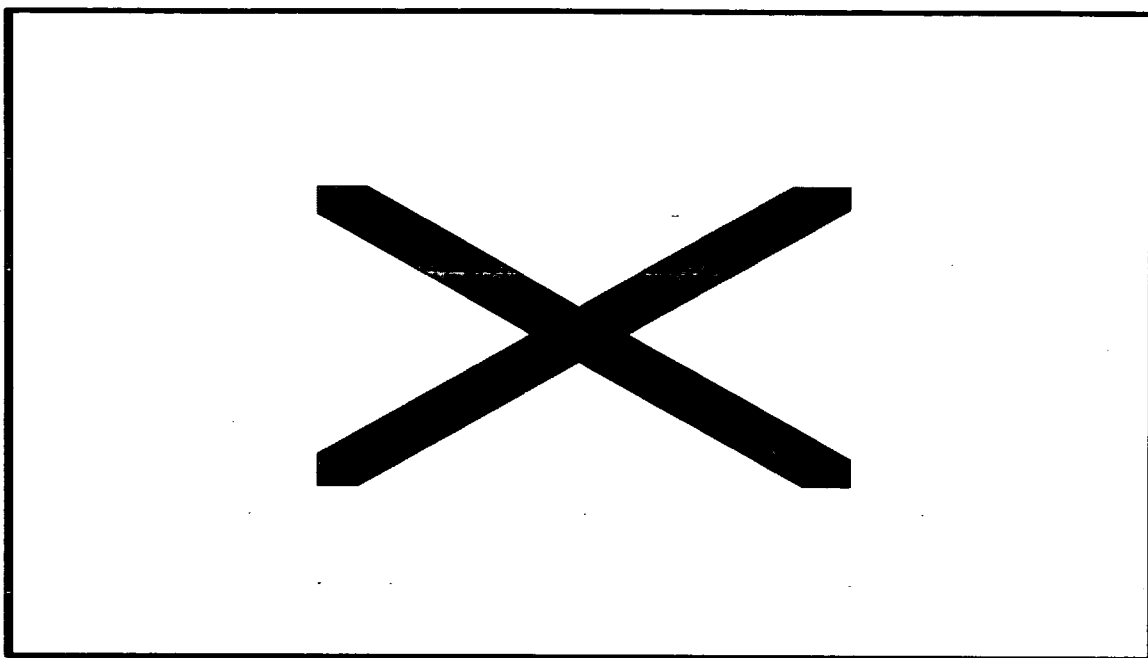
sensor surface to be contacted with a fluid whose property is to be measured, the sensor surface has an adherent non-porous coating of a poly-oligomer containing fluorine



**FIG. 6**



**FIG. 3**



**FIG. 4**